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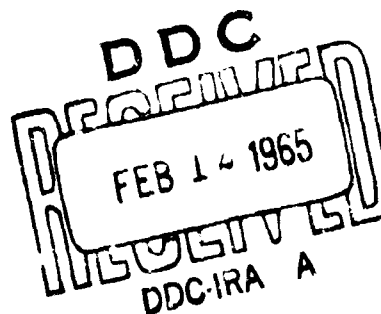
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ALKALI METAL RESISTANT INSULATION

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USAF Contract AF33(615)1350

Project No. 8128  
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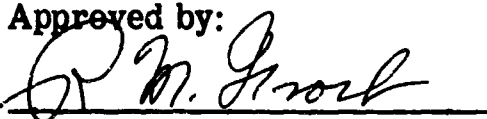
**HIGH TEMPERATURE  
ALKALI METAL RESISTANT INSULATION**

**3rd Quarterly Progress Report  
Contract AF33(615)1360, Task No. 8128-06**

**January 10, 1965**

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## NOTICE

The work covered by this report was accomplished under Air Force Contract AF33(615)1360, but this report is being published and distributed prior to Air Force review. The publication of this report, therefore, does not constitute approval by the Air Force of the findings and conclusions contained herein. It is published for the exchange and stimulation of ideas.

## **FOREWORD**

**This 3rd Quarterly Report is submitted by the Aerospace Electrical Division, Westinghouse Electric Corporation, Lima, Ohio, on Air Force Contract AF33(615)1360, Project No. 8128, Task No. 8128-06 High Temperature Alkali Metal Resistant Insulation. The contract is administered by the Air Force Aero-Propulsion Laboratory, Research and Technology Division, Wright-Patterson Air Force Base, Dayton, Ohio. Mr. Lester Schott is Project Engineer for APIP on this contract.**

**The work described in this report was done by personnel in the Materials Development Section and Research Laboratory at Westinghouse AED, Lima, Ohio and Westinghouse Research and Development Center, Churchillborough, Pennsylvania.**

**The participation of the following is specifically acknowledged: W. H. Snavely, Project Engineer; Dr. R. J. Towner and R. E. Stapleton, Composite Seals; J. W. Ogden, Transformer Design and Testing; R. E. McVay, Magnetics; L. D. Fraley, Stress Analysis; Dr. C. Hirayama, Active Metal Seals and D. J. Boes, Potassium Vapor Corrosion Tests.**

## **ABSTRACT**

**This report covers the progress during the Third Quarter on Air Force Contract AF33(615)1360.**

**Five-hundred hour corrosion tests in 850 C potassium vapor have been completed on additional materials. Ceramic-to-metal seals were fabricated by hot pressing techniques and also by active metal brazing. Electrical testing of the active metal braze seal in potassium vapor was conducted at 600 C. A preliminary test transformer constructed of potassium resistant materials was electrically tested in argon at three different temperatures up to 600 C. The same transformer was electrically energized and tested in potassium vapor at room temperature, 290, 330 and 450 C.**

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## I. INTRODUCTION

This report covers the third quarter from October 1, 1964 to December 31, 1964 on Air Force Contract AF33(615)1360, High Temperature Alkali Metal Insulation.

The program was initiated to investigate the effect of 600-850 C potassium vapor on selected materials in an electrical test device. The test device is a transformer designed to operate in potassium vapor at 600 C. Various voltages to 1000 Vac and frequencies to 3200 cps are to be applied to the primary windings. Fifty amperes at thirty-two volts will be one output of the secondary winding. The transformer is to be operated at these electrical test conditions for 500 hours. Examination of the materials after the test period will be done to determine the effect of the combined environmental test conditions.

## II. SUMMARY OF WORK PERFORMED AND MAJOR RESULTS

1. Potassium vapor corrosion tests run at 850 C were completed.
2. An electrical terminal seal was charged with purified potassium. The electrical resistance changes of the Lucalox ceramic insulator, due to potassium vapor, were determined to a temperature of 600 C.
3. Concentric ring seals have been hot pressed to approximately 100 percent density with strong interface bonding and a high degree of concentric symmetry. These results were obtained with a number of different refractory metals in combination with high purity ( > 99.9%  $\text{Al}_2\text{O}_3$ ) alumina.
4. Thermal expansion mismatch has been experimentally found to be the determinant factor in achieving crack-free ring seal specimens. The columbium-alumina seal can be reproducibly made without cracking. All other refractory metal combinations have caused radial cracks to appear in the alumina.
5. Hot pressed concentric ring seals (after surface lapping) were helium leak tested at room temperature. Columbium seals were found to have no measurable leak. A molybdenum seal was also leak tight, although the alumina insulator was cracked. Tantalum seals showed a significant leak rate.
6. A hot-pressed five-layer end-to-end seal composed of molybdenum, molybdenum and alumina and pure alumina was found to have a

modulus of rupture of 32, 300 psi. Cold-pressed component parts have been made for additional testing.

7. Specimens of all hot-pressed refractory metal-alumina combinations have been lapped, polished and etched for metallographic studies.
8. Alumina insulation used in the concentric seal configuration was hot-pressed as individual wafers. This material was found to have an electrical resistivity in the same decade range at 850 C as high purity sapphire. The disks were translucent, possessed a characteristic grain structure, and were close to theoretical density.
9. Tests on magnetic materials were conducted after exposure to potassium vapor at 600 C for 500 hours. Potassium vapor exposure did not significantly change the a-c core loss or d-c magnetization properties of Cubex, Hiperco 27, or Armco Ingot Iron at the proposed operating flux density of 80 KL/in<sup>2</sup>. The 5% Al-Fe alloy and 3% Al, 1% Y-Fe alloy a-c core loss properties show no improvement over Cubex at 400 cps.
10. Methods of producing a pore-free nickel plating to protect the silver core of nickel-clad silver conductors have been developed.
11. Nickel-clad silver conductors have been plated with nickel by this technique and have resisted corrosive attack by 600 C potassium vapor for 500 hours.
12. Columbium-1% zirconium clad dispersion strengthened copper conductors with a 28 percent cladding have been fabricated.

13. A No. 22 AWG float-zone refined rhodium conductor has been exposed to 850 C potassium vapor for 172 hours. The hardness decreased but very little change in weight was observed.
14. The preliminary test transformer has been electrically tested in saturated potassium vapor at temperatures to 450 C. An electrical open circuit occurred above this temperature. The active metal brazed seal was still leak tight after testing.
15. The ceramic coil form transformer has been fabricated and electrically tested at room temperature. A temperature rise of 125 C was observed at 1000 volts and 400 cps input with a 50 ampere output when the secondary winding of the transformer was connected to a resistive load.

### III. EXPERIMENTAL WORK

#### A. Potassium Vapor Exposure Tests

##### 1. Results of Additional 850 C Potassium Vapor Exposure Tests

The materials listed in Table I were exposed to potassium vapor at 850 C for the purpose of evaluating their corrosion resistance. All the materials were exposed for 500 hours, with the exception of the No. 22 AWG rhodium conductor and the molybdenum, molybdenum-alumina, alumina hot-pressed composite. These samples were exposed to 850 C potassium vapor for 172 and 330 hours respectively. Weight change data given in Table I shows the fused fluoride eutectic underwent a considerable reduction in weight. The shape of the fluoride test piece was not altered appreciably considering the weight change. As Table I shows, the barium fluoride is attacked more than the calcium fluoride. The conclusion is that high purity potassium preferentially removed the barium fluoride from the eutectic at the temperature of 850 C. The eutectic still possessed a coherent structure so it has been selected as a potting material for use at 600 C. The hot-pressed molybdenum composite had good resistance to potassium vapor. The alumina portion of this composite was still bonded firmly to the molybdenum-alumina cermet. Small cracks which were present in the alumina before exposure became more visible.

TABLE I. 850 C Potassium Vapor Corrosion Test Data - Exposure Time 500 Hours

No. Designation	Initial Wt. #	Final Wt. #	Difference #	Potassium Charge #	Initial Appearance	Final Appearance
1. Rhodium	0.3696	0.3707	+0.0011	0.611	Silver wire	Darkened to dull grey
2. Si <sub>3</sub> N <sub>4</sub>	0.5731	--	--	0.569	Light grey rocker	Disintegrated
3. LiF	1.4018	--	--	0.623	Clear glass rectangle	Sample melted
4. CaF	1.0299	0.9308	-0.0991	0.586	Clear glass chip	Pink color
5. BaF	1.1718	0.0609	-1.1209	0.616	Clear glass chip	Yellow - chip
6. CaF <sub>2</sub> BaF <sub>2</sub>	0.9588	0.5176	-0.4412	0.495	Grey triangle	Porous
7. Mo-M-Al <sub>2</sub> O <sub>3</sub>	2.0963	2.1170	+0.0207	0.810	Grey pellet	Bottom layer turned black & swelled 0.007 inch
8. Al <sub>2</sub> O <sub>3</sub> -610	0.9294	0.9315	+0.0021	0.594	Milk white rocker	Turned light grey
9. Blank	No sample exposed in this capsule			0.281		
10. Al <sub>2</sub> O <sub>3</sub> -610	0.5437	0.5438	+0.0001	0.473	Milk white rocker	Light grey

\*Off test after 172 hours

\*\*Off test after 330 hours

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## **B. Ceramic to Metal Seals**

### **1. Electrical Testing in 600 C Potassium Vapor**

An electrical test capsule was made by using an active metal braze (60% titanium, 25% nickel, 15% columbium) to join a short piece (~ 2 inches) of 5/8 inch O. D. Lucalox between two columbium metal pieces. A photograph of this type seal is in the Second Quarterly Report on page 23.

The test capsule was charged with high-purity potassium (99.9%), evacuated and closed by tungsten inert-gas welding. A furnace was constructed and insulated with Fiberfrax to keep the capsule at a uniform temperature. The capsule furnace and electrical leads were then placed inside the bell jar of a vacuum system.

The two capsule leads were connected inside the bell jar to electrical feed-through insulators. The system was evacuated to  $10^{-5}$  torr. Resistance readings were then taken with a Freed megohmmeter to a temperature of 475 C where the 500 volts output from the megohmmeter caused breakdown of the vapor absorbed on the insulator walls. At this point, the resistance reading was  $1.35 \times 10^5$  ohms. A Triplett volt-ohm-millimeter was used from 475 C to 600 C to determine resistance. The resistance readings versus temperature readings are shown in Figure 1.

Voltage breakdown measurements were taken at 493 C using a Slaughter leakage tester. The reading obtained was 4.5 milliamperes at 275 Vac.

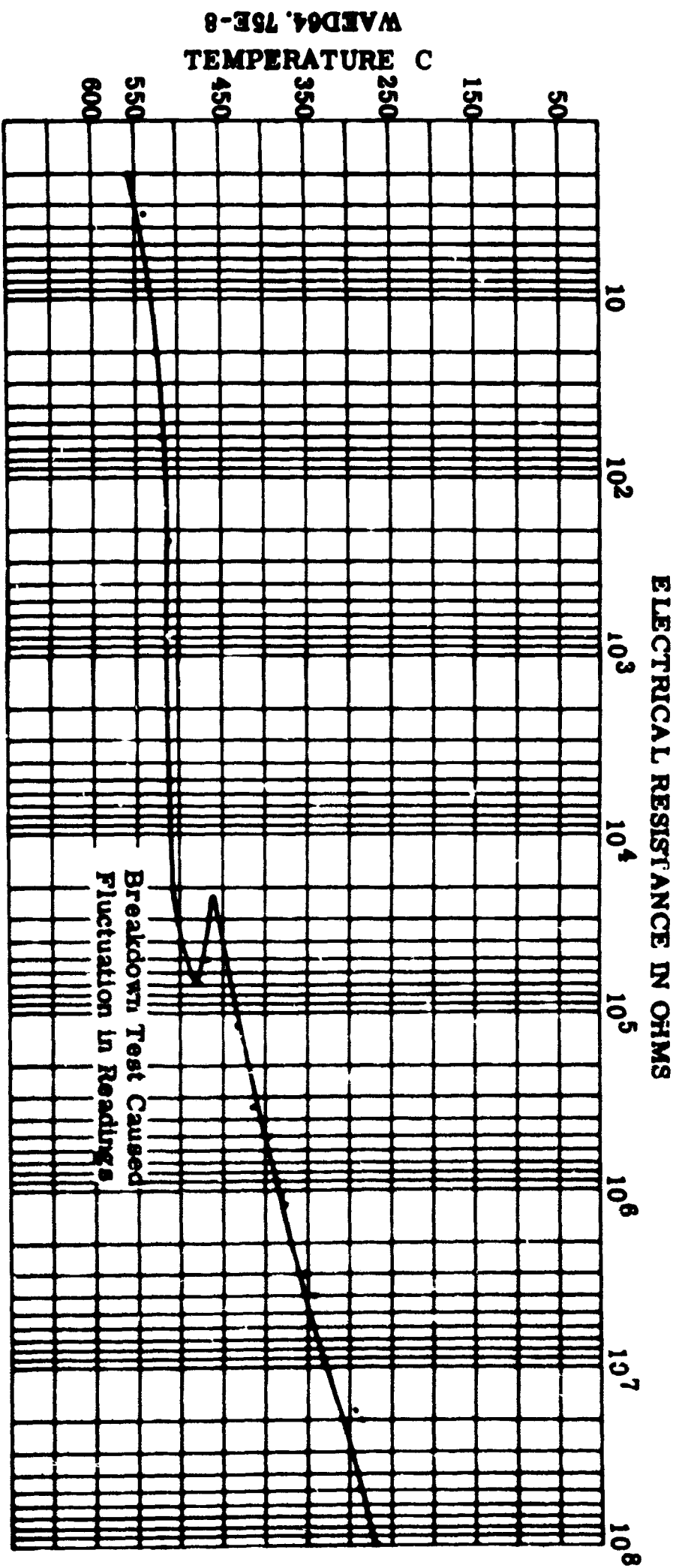


FIGURE 1. Electrical Resistivity of Ceramic-to-Metal Terminal Seal  
During Potassium Vapor Exposure

This test capsule was exposed for 24 hours at 600 C, then taken from the vacuum furnace and cut open in an argon-filled glove box. It was noted at this time that the potassium level had contacted the bottom edge of the Lucalox insulator. Potassium may have crept up the ceramic wall at high temperature, causing the low resistance reading at 600 C ( $\sim$ 2.04 ohms).

## 2. Pressure Sintered Ceramic-Metal Composites and Hermetic Seals

Work continued on the development of hermetic potassium resistant electrical terminal seals by hot pressing and sintering metal-to-ceramic compacts. The hot pressing operation was applied to compacts of metals, 50 v/o metal + 50 v/o alumina mixtures, and alumina which had low initial densities. This provided composite pellets with strong bonds across the various interfaces.

### a. Materials Used in Composites

The powders used to date in this investigation were as follows:

<u>Powder</u>	<u>Purity</u>	<u>Particle Size</u>	<u>Source</u>
Molybdenum	> 99.5%	-150+325 mesh	Plasmadyne
Tungsten	> 99.9%	3-5 microns	Engineered Materials
Tantalum	> 99.5%	-325 mesh	Fansteel
Columbium	> 99.5%	-80+325 mesh	Fansteel
Alumina	> 99.9% Metallurgical Grade Linde A	0.3 microns	Union Carbide

### b. Concentric Ring Seals - Cold and Hot Pressed

Dies were designed and constructed from hardened tool steel ( $R_c 60$ ) for cold compacting 50 percent dense ring-shaped pellets of metal, metal + ceramic, and ceramic

powders. After cold compacting, the pellets were assembled into a concentric configuration and hot pressed simultaneously in the axial direction to full density. The approximate diameters of the cold compacted pellets, based on die dimensions are given below:

<u>Powder Material</u>	<u>Die Set #</u>	<u>Compact Condition</u>	<u>Compact Size</u>		<u>Compact Shape</u>
			<u>O. D. , in</u>	<u>I. D. , in</u>	
Metal	1	Green	1. 376	1 136	Ring
Metal + Ceramic	2	Green	1. 126	0. 881	Ring
Ceramic	3	Green	0. 937	0. 413	Ring
Ceramic	3	*Pre-Sintered	0. 876*	0. 386*	Ring
Metal + Ceramic	4	Green	0. 376	0. 136	Ring
Metal	5	Green	0. 131	--	Cylinder

\*Note: Compact size after shrinkage during pre-sintering

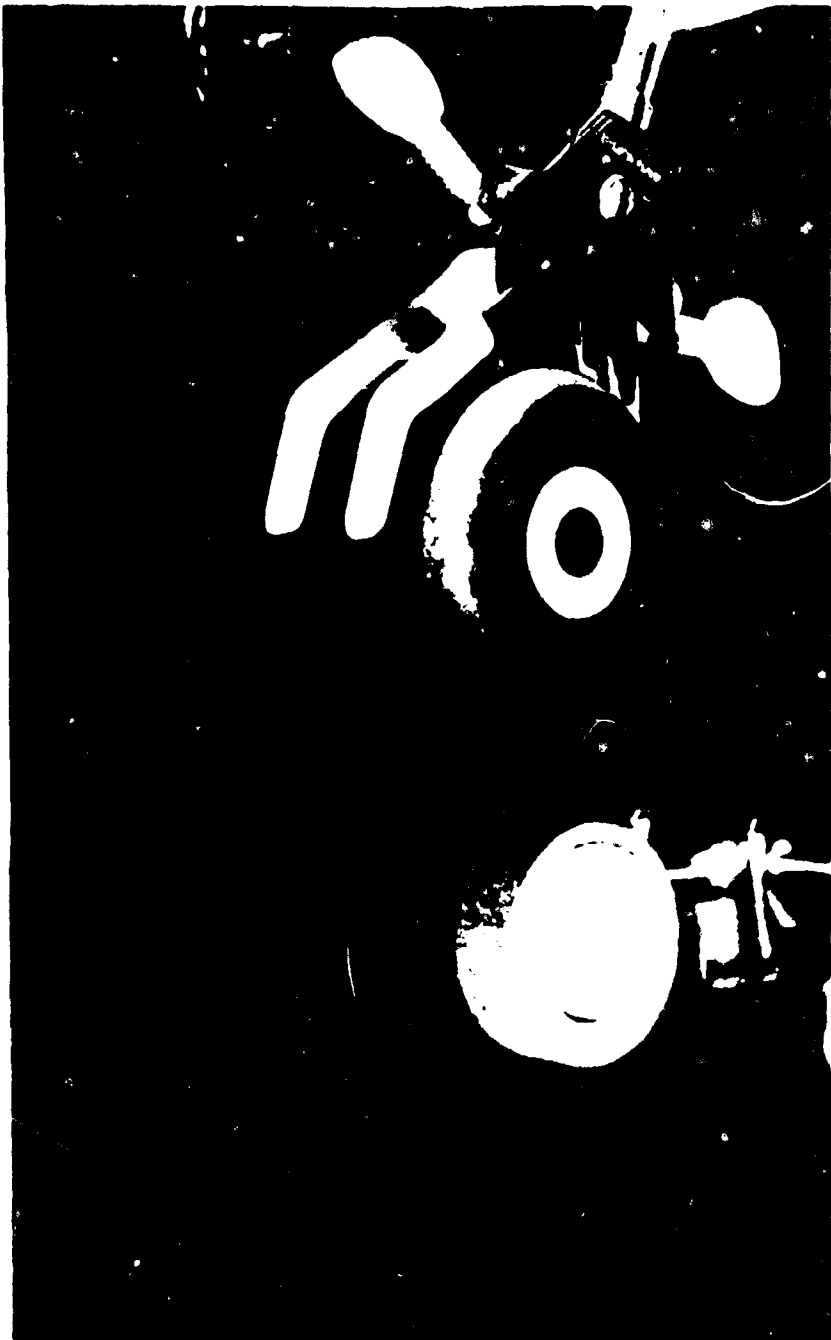
All of the ring-shaped dies were designed with a 0.002 inch taper on the diameter per one inch of compact length for ease of ejection. Compact lengths of up to one inch were provided for in the die design. The actual compacts used for hot pressing had lengths of either 0.5 inch or one inch. The desired density of the compacts was attained by controlling the weight of powder charge and length of compact. The density of the alumina compacts was further controlled by the drying and pre-sintering conditions. It was generally impossible to make precise measurements on the

dimensions and density of the green compacts because of their fragility. However, in those cases where measurements were made, there was good agreement with those intended. The photograph in Figure 2 illustrates the typical size and geometry of compacts before and after hot pressing to a length one half the original.

The compacted metal-to-ceramic seals were made in dies (1.385 in. I. D.) machined from Speer Carbon Co. high purity graphite. Figure 3 shows a photograph of the hot pressing fixture which was constructed to compact the larger diameter ring seals. The essential features of the hot pressing fixture described in the Second Quarterly Report have been incorporated in the new design shown in Figure 3.

A number of refinements have been made, however, which have resulted in improved operating characteristics. Six ring seals were hot pressed in this fixture without the necessity of replacing any of its components. Better argon circulation around the graphite parts has been achieved by strategic location of inlet and outlet tubes. A mullite outer protection tube has replaced the alumina tube previously used.

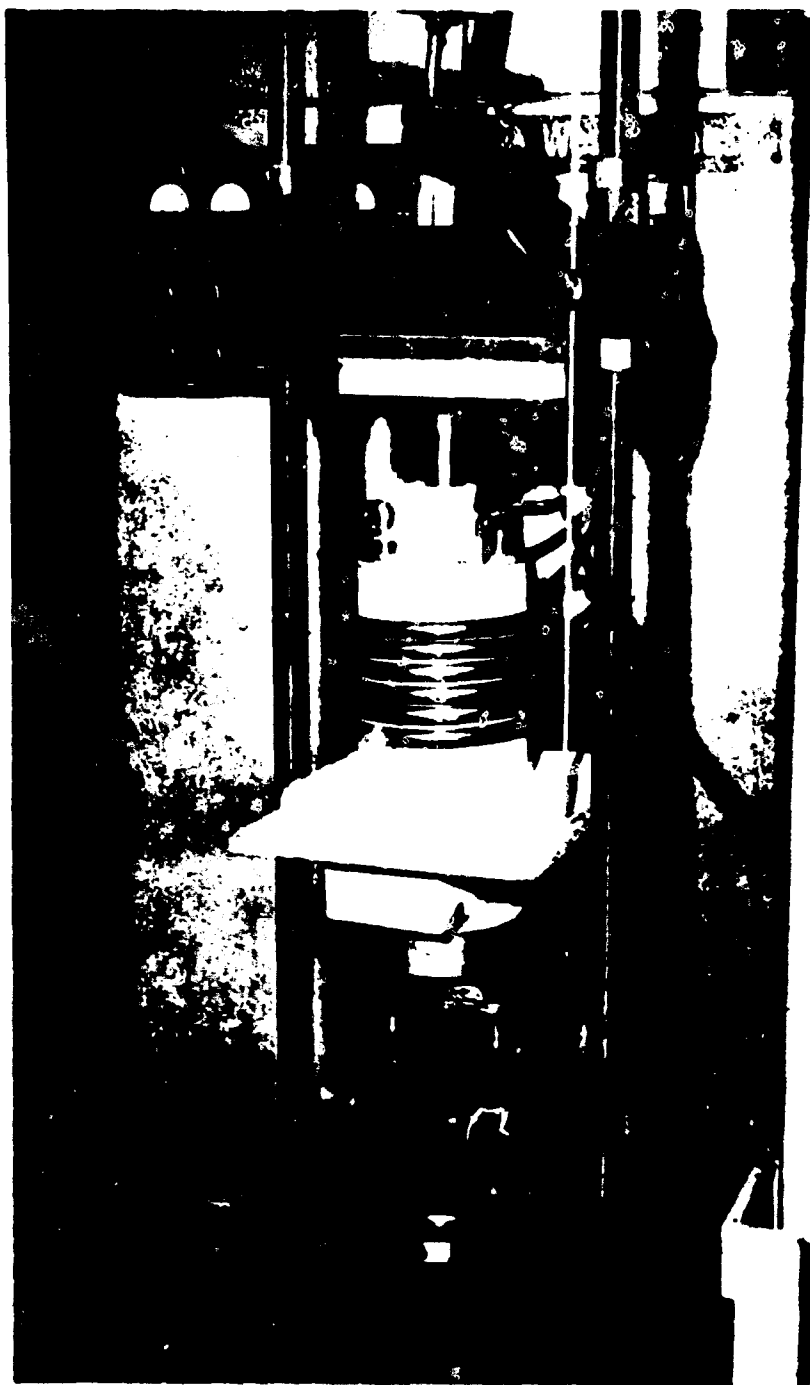
The seals of the concentric ring configuration, which have been made since the Second Quarterly Report, as well as



After Hot Pressing

Before Hot Pressing

FIGURE 2. Size and Geometry of Concentric Ring Seal Compacts Before and After Hot Pressing



**FIGURE 3. Hot Pressing Fixture Used to Compact Large Diameter Concentric Ring Seals**

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those of the smaller diameter cylindrical end-to-end configuration which will be discussed later, are listed in Table II.

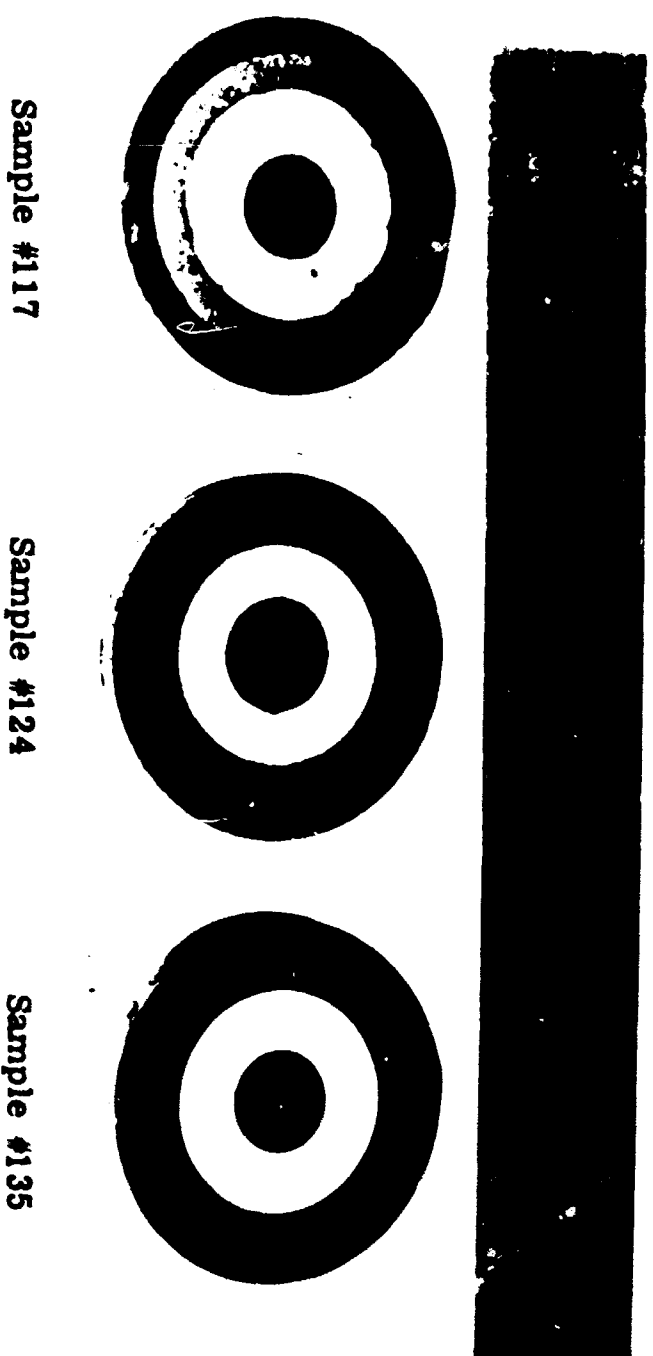
The photograph in Figure 4 shows the Mo-Al<sub>2</sub>O<sub>3</sub> (sample #117), Ta-Al<sub>2</sub>O<sub>3</sub> (sample #124) and Cb-Al<sub>2</sub>O<sub>3</sub> (sample #135) hot pressed seals. Unlike the cylindrical end-to-end configuration hot pressed pellets made earlier, both Mo-Al<sub>2</sub>O<sub>3</sub> ring seals (samples #117 and #118) contained fine cracks, mainly in the radial direction, which in some instances traversed completely through the center of the seal and terminated only in the outermost metal ring. Likewise, the Ta-Al<sub>2</sub>O<sub>3</sub> ring seals (samples #124 and #129) contained radial cracks. These cracks were believed to be due to the differences in coefficient of thermal expansion of the metal (molybdenum or tantalum) and alumina, and were aggravated by the relatively large diameter of the hot pressed seal, 1.38 inches O. D. The two Cb-Al<sub>2</sub>O<sub>3</sub> ring seals (samples #135 and #140) were free from cracks. Maintenance of concentricity, and achievement of high density and bonding between components were very satisfactory in all six of the ring seals.

TABLE II. Hot Compacted Metal-to-Ceramic Beals

Beal Configuration	Sample No.	No. of Layers	Materials						Size of Hot Pressed Beal, Inches		Hot Pressing Conditions (a)		
			Metal	Mixture	Ceramic	Mixture	Metal	Thk.	Length	Temp. °C	Time, Min.	Pressure, psi	
Cylindrical End to End	56	5	Mo (98%) <sup>a</sup>	Mo-Al <sub>2</sub> O <sub>3</sub> (98%) <sup>a</sup>	Al <sub>2</sub> O <sub>3</sub> (99.9%) <sup>b</sup>	Mo-Al <sub>2</sub> O <sub>3</sub> (98%) <sup>a</sup>	Mo (98%) <sup>a</sup>	0.371	0.506	1800±	40	5,000	1548
Cylindrical End to End	57	5	Mo (50%) <sup>a</sup>	Mo-Al <sub>2</sub> O <sub>3</sub> (50%) <sup>a</sup>	Al <sub>2</sub> O <sub>3</sub> (98%) <sup>a</sup>	Mo-Al <sub>2</sub> O <sub>3</sub> (50%) <sup>a</sup>	Mo (98%) <sup>a</sup>	0.204	0.504	1800±	60	5,000	1505
Cylindrical End to End	58	3	Mo (98%) <sup>a</sup>	Mo-Al <sub>2</sub> O <sub>3</sub> (98%) <sup>a</sup>	Al <sub>2</sub> O <sub>3</sub> (98%) <sup>a</sup>	---	---	0.20	0.50	1800±	61	5,000	1571
Concentric Ring (Outside to Center)	117	5	Mo (98.9%), 48.9% <sup>a, c</sup>	Mo-Al <sub>2</sub> O <sub>3</sub> (98.9%) <sup>a</sup>	Al <sub>2</sub> O <sub>3</sub> (98%) <sup>a, c</sup>	Mo-Al <sub>2</sub> O <sub>3</sub> (98%) <sup>a</sup>	Mo (98%) <sup>a</sup>	1.20	0.40	1800±	97	5,000	1564
Concentric Ring (Outside to Center)	118	5	Mo (98.9%), 48.9% <sup>a, c</sup>	Mo-Al <sub>2</sub> O <sub>3</sub> (98%) <sup>a</sup>	Al <sub>2</sub> O <sub>3</sub> (98%) <sup>a, c</sup>	Mo-Al <sub>2</sub> O <sub>3</sub> (98%) <sup>a</sup>	Mo (98%) <sup>a</sup>	1.20	0.40	1800±	70	5,000	1560
Concentric Ring (Outside to Center)	124	5	Ta (98%) <sup>a</sup>	Ta-Al <sub>2</sub> O <sub>3</sub> (98%) <sup>a</sup>	Al <sub>2</sub> O <sub>3</sub> (98%) <sup>a</sup>	Ta-Al <sub>2</sub> O <sub>3</sub> (98%) <sup>a</sup>	Ta (98%) <sup>a</sup>	1.20	0.20	1800±	100	5,000	1500
Concentric Ring (Outside to Center)	129	5	Ta (98%) <sup>a</sup>	Ta-Al <sub>2</sub> O <sub>3</sub> (98%) <sup>a</sup>	Al <sub>2</sub> O <sub>3</sub> (98%) <sup>a</sup>	Ta-Al <sub>2</sub> O <sub>3</sub> (98%) <sup>a</sup>	Ta (98%) <sup>a</sup>	1.20	0.20	1800±	40	5,000	1460
Concentric Ring (Outside to Center)	128	5	Co (70%) <sup>a</sup>	Co-Al <sub>2</sub> O <sub>3</sub> (70%) <sup>a</sup>	Al <sub>2</sub> O <sub>3</sub> (98%) <sup>a</sup>	Co-Al <sub>2</sub> O <sub>3</sub> (70%) <sup>a</sup>	Co (98%) <sup>a</sup>	1.20	0.204	1800±	97	5,000	1460
Concentric Ring (Outside to Center)	140	5	Co (70%) <sup>a</sup>	Co-Al <sub>2</sub> O <sub>3</sub> (70%) <sup>a</sup>	Al <sub>2</sub> O <sub>3</sub> (98%) <sup>a</sup>	Co-Al <sub>2</sub> O <sub>3</sub> (70%) <sup>a</sup>	Co (98%) <sup>a</sup>	1.20	0.203	1800±	77	5,000	1604

NOTES:

- Densify before hot compacting (estimated from powder charge weight and die volume).
- Densify before hot compacting (measured).
- Two 0.10 inch long pellets were stacked end-to-end for hot pressing.
- Lower powder pressed into smaller cylindrical hole - not cold compacted.
- Pressure of 1000 psi was applied and maintained at all temperatures above 1000°C. Temperature was held near the maximum listed for 30-60 minutes.



Note: Outer ring - metal, cermet ring, ceramic, cermet metal - center piece.

FIGURE 4. Mo-Al<sub>2</sub>O<sub>3</sub> (Sample #117), Ta-Al<sub>2</sub>O<sub>3</sub> (Sample #124) and Cb-Al<sub>2</sub>O<sub>3</sub> (Sample #135) Hot Pressed Concentric Ring Seals

c. Leak Testing of Concentric Ring Seals

The following seals were tested at room temperature in a Veeco Leak Detector MS-9 with one side exposed to 95% N<sub>2</sub>+5% He flowing from a probe, while the other side was under a vacuum of less than 10<sup>-5</sup> torr. The following results were obtained by comparing with a standardized system leak rate:

Sample No.	Seal Type	Size of Seal, Inches		Degree of Leaking Detected	Remarks
		Dia.	Length		
117	Mo-Al <sub>2</sub> O <sub>3</sub>	1.38	0.49	None	Seal contained cracks
124	Ta-Al <sub>2</sub> O <sub>3</sub>	1.38	0.25	Excessive	Seal contained cracks
129	Ta-Al <sub>2</sub> O <sub>3</sub>	1.38	0.25	Excessive	Seal contained cracks
135	Cb-Al <sub>2</sub> O <sub>3</sub>	1.38	0.256	None	Seal uncracked
140	Cb-Al <sub>2</sub> O <sub>3</sub>	1.38	0.262	None	Seal uncracked

The above data indicate that leakage was not detectable for seals #117, #135, and #140. Although the Mo-Al<sub>2</sub>O<sub>3</sub> seal contained cracks, it was leak tight while the two Ta-Al<sub>2</sub>O<sub>3</sub> seals were not. Perhaps this is related to the greater length of the Mo-Al<sub>2</sub>O<sub>3</sub> seal. Sample #124 was found to have an excessive leak rate at radial cracks with the helium probe, but the interfaces between components away from the crack appeared not to leak. On the other hand, sample #129 showed a high-leak rate at the alumina and the inner

tantalum cermet interface. The seal did not appear to leak in the vicinity of cracks.

d. Cylindrical End-to-End Seals

(1) Hot Pressing

Three more cylindrical end-to-end seals of the Mo-Al<sub>2</sub>O<sub>3</sub> type (samples #56, #57, and #58 as shown in Table II) were hot pressed in three-layer and five-layer configurations. These were of a form suitable for determination of modulus of rupture and corrosion resistance in saturated potassium vapor. However, only a modulus of rupture test was actually made and the plans to fabricate Ta-Al<sub>2</sub>O<sub>3</sub> seals of this configuration were abandoned due to the cracking encountered in the concentric ring seals of Mo-Al<sub>2</sub>O<sub>3</sub> and Ta-Al<sub>2</sub>O<sub>3</sub>. It was decided to concentrate efforts instead on production of ring seals of Cb-Al<sub>2</sub>O<sub>3</sub> which did not crack.

(2) Modulus of Rupture

The modulus of rupture was determined to be 32,300 psi in three-point loading for a five-layer cylindrical end-to-end seal of Mo-Al<sub>2</sub>O<sub>3</sub> (sample #57) whose fabricating conditions are given in Table II. This value may be compared with a modulus of rupture of 14,500 psi for the three-layer Mo-Al<sub>2</sub>O<sub>3</sub> seal (sample #18) described in the Second Quarterly Report. Photographs

showing the fractured ends of sample #57 are presented in Figure 5. The fracture occurred in the  $\text{Al}_2\text{O}_3$  layer and not at the cermet- $\text{Al}_2\text{O}_3$  interface.

(3) Potassium Exposure Tests

One end of a fractured Mo- $\text{Al}_2\text{O}_3$  seal (sample #18) containing only the 50 v/o Mo + 50 v/o  $\text{Al}_2\text{O}_3$  layers, after it was broken in a modulus of rupture test, was exposed to saturated potassium vapor at 850 C for approximately 330 hours. As previously described in Section III. A. 1. , no changes were observed visually.

(4) Microstructure

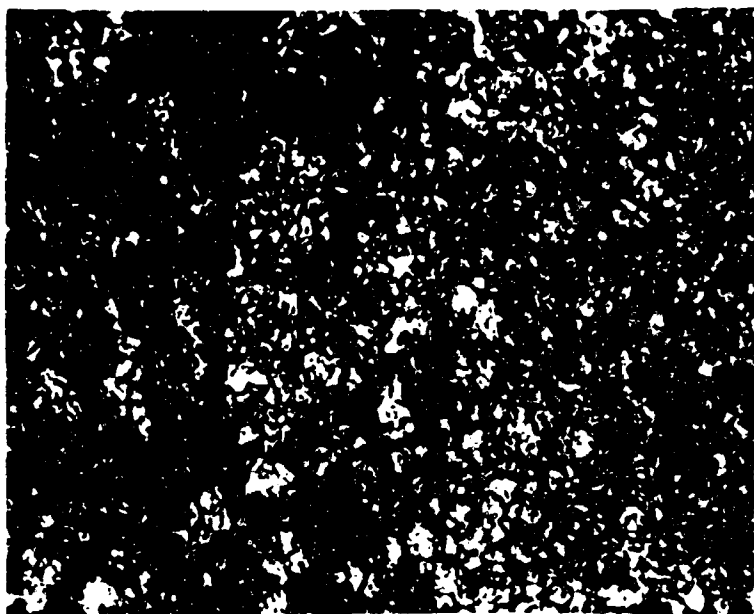
The microstructures of the three-layer tungsten -  $\text{Al}_2\text{O}_3$  seal (sample #21 in Table V of the Second Quarterly Report) and of the five-layer Mo- $\text{Al}_2\text{O}_3$  seal (sample #57 in Table II of this report) at the metal-cermet interfaces are presented in Figures 6 and 7. The dispersion of the metal and alumina phases was quite uniform in the cermet layer and good bonding was achieved at all interfaces. Both the tungsten and molybdenum recrystallized during hot pressing.

3. Stress Analysis of Concentric Ring Seals

Visual inspection of concentric ring seals made of molybdenum and alumina and tantalum and alumina revealed radial cracks running through



FIGURE 5. Fractured Ends of Sample #57 After Modulus of Rupture Tests



Etched  
Sample #21

500X  
Longitudinal Section

FIGURE 6. Interface Between 50 v/o Tungsten + 50 v/o Al<sub>2</sub>O<sub>3</sub> Mixture (left) and Tungsten (right)



Etched  
Sample #57

500X  
Longitudinal Section

FIGURE 7. Interface Between 50 v/o Mo + 50 v/o Al<sub>2</sub>O<sub>3</sub> Mixture (left) and Mo (right)

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the rings and terminating at the outer metal ring. It is surmised that the crack was the result of a high tensile hoop stress in the cermet adjacent to the outer ring when the seal cools from the hot pressed temperature. The following calculations were made to approximate the magnitude of the stress and determine the effect of the principal variables.

Let: Subscript c refer to the outer cermet ring.

Subscript m refer to the outer metal ring.

The thermal expansion mismatch at the common radius (R) is:

$$\Delta = \alpha_c R \Delta T - \alpha_m R \Delta T$$

where:

$\Delta T$  = (Temperature at which pieces are joined) —  
(room temperature)

$\alpha$  = Coefficient of thermal expansion.

This mismatch in expansion is accommodated by deflection of the two rings:

$$\Delta = \Delta_c + (-) \Delta_m$$

$$\Delta = R \frac{S_c}{E_c} - R \frac{S_m}{E_m}$$

$$\Delta = R \frac{PR}{E_c t_c} + R \frac{PR}{E_m t_m}$$

where:

P = pressure at interface (+ @c) and (-@m)

Combining (1) and (2):

$$P = \frac{(\alpha_c - \alpha_m) \Delta T}{R \left( \frac{1}{E_c t_c} + \frac{1}{E_m t_m} \right)}$$

and

$$S_c = \frac{PR}{t_c} = \frac{(\alpha_c - \alpha_m) \Delta T}{\frac{1}{E_c} + \frac{1}{E_m} \frac{t_c}{t_m}}$$

where:

$S_c$  = hoop stress in cermet

$t_c, t_m$  = thickness of rings

$E_c, E_m$  = modulus of elasticity.

Assuming equal radial thickness, the following values were calculated for various metals used in a 50 v/o metal + 50 v/o alumina cermet:

<u>Metal Component in Cermet</u>	<u><math>S_c</math> (psi)</u>
Mo	47,600
Ta	12,300
Cb	2,300

These calculations indicate that a much lower residual tensile stress will result at room temperature if columbium is used.

Therefore, columbium is the preferred metal, from a mechanical standpoint, in a concentric ring seal over molybdenum or tantalum.

The values used to arrive at the hoop stress ( $S_c$ ) figures are given below:

<u>Material</u>	<u>Modulus of Elasticity at Room Temperature E (<math>10^6</math> psi)</u>	<u>Coefficient of Thermal Expansion, at Room Temperature, <math>\alpha</math> (<math>10^{-6}</math> per <math>^{\circ}\text{C}</math>)</u>
$\text{Al}_2\text{O}_3$	55	7.5
Mo	47	4.9
50 v/o Mo + 50 v/o $\text{Al}_2\text{O}_3$	51*	6.2*
Ta	27	6.5
50 v/o Ta + 50 v/o $\text{Al}_2\text{O}_3$	41*	7.0*
Cb	15	7.2
50 v/o Cb + 50 v/o $\text{Al}_2\text{O}_3$	35*	7.35*

\*Estimated value representing the average of the individual values for metal and  $\text{Al}_2\text{O}_3$ .

#### 4. Pressure Sintered Insulators Representative of Composite Seals

A series of individual insulator test samples were prepared using similar methods and the same raw material as the alumina insulation incorporated in the concentric ring seal. These specimens were prepared to measure the electrical properties of the insulation independent of refractory metal effects. In addition, microstructure specimens can be more conveniently prepared.

Powdered alumina (Linde A,  $> 99.9\%$   $\text{Al}_2\text{O}_3$ ) was mixed with an organic binder to obtain green strength after dry pressing. A 10% water solution of Elvanol (DuPont #75-05) was used resulting in a dry press mixture containing 3% binder and approximately 0.5% moisture.

Thin disks were dry pressed (0.444 inch diameter x 0.035 inch thick) with this mix at 20,000 psi in a hardened steel die. After pressing and oven drying, the disks were pre-fired at 1288 C in a Globar tube furnace. This treatment removed the organic binder and pre-sintered the disks to achieve sufficient strength for subsequent handling.

Diameter shrinkages were determined after sintering and averaged about 7%. On the basis of weight and volume measurements, the disks were approximately 50% of theoretical density.

The pre-sintered specimens were stacked in a graphite die (re-bored to obtain a clean and uniform I. D.) with powdered graphite spacers between disks. The die was inductively heated to 1538 C for 30 minutes at 5,000 psi applied pressure. After cooling, the pressed compact was ejected and the alumina disks were separated from their graphite spacers.

The disks were cleaned and lapped; after this treatment they appeared optically translucent. Density measurements were made

by weighing the samples in air and in water. These results indicate the specimens are very close to theoretical density. However, this test is not precise because the sample weights were very small and weight differences were in the third decimal place.

a. Microstructure

A number of hot pressed disks were polished with diamond abrasives and etched in boiling, concentrated sulfuric acid (3-7 minutes) to reveal grain structure. A series of representative micrographs are shown in Figure 8 at three different magnifications. These pictures show that some secondary recrystallization (discontinuous or exaggerated grain growth) has occurred. The overall grain size appears to be smaller than Lucalox and distinctly different in shape. Figure 9 shows a sample of Lucalox prepared in a similar manner by polishing and etching in boiling sulfuric acid. Lucalox grains are essentially equiaxed whereas the hot pressed aluminum oxide sample consists of long, thin crystallites. The long axis of each grain appears to be aligned perpendicular to the direction of applied pressure.

Figure 10 shows a polished and etched specimen of Triangle RR (+99.7% recrystallized alumina). The grains are shaped similarly to Lucalox; however, a number of pores



1000X

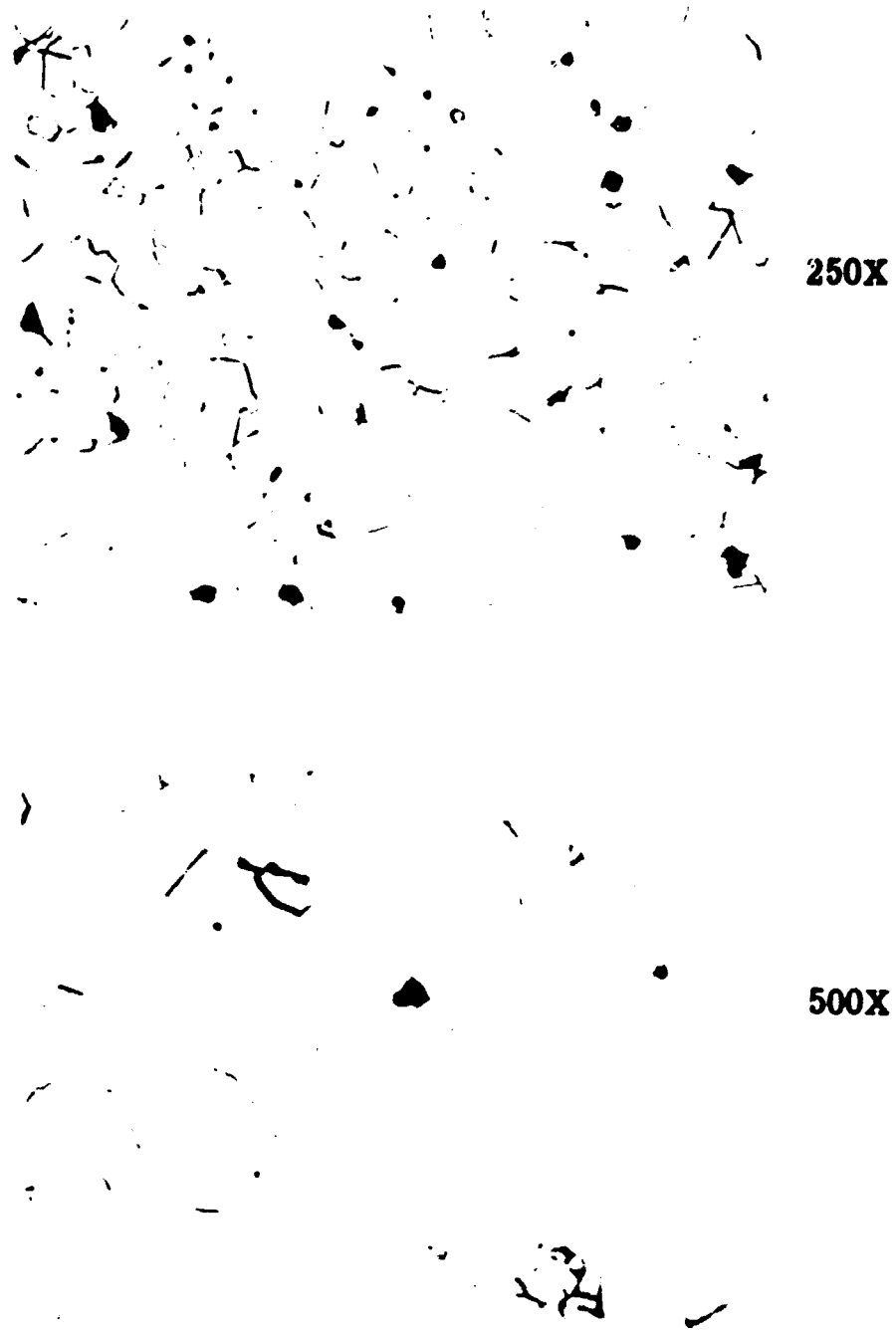


500X



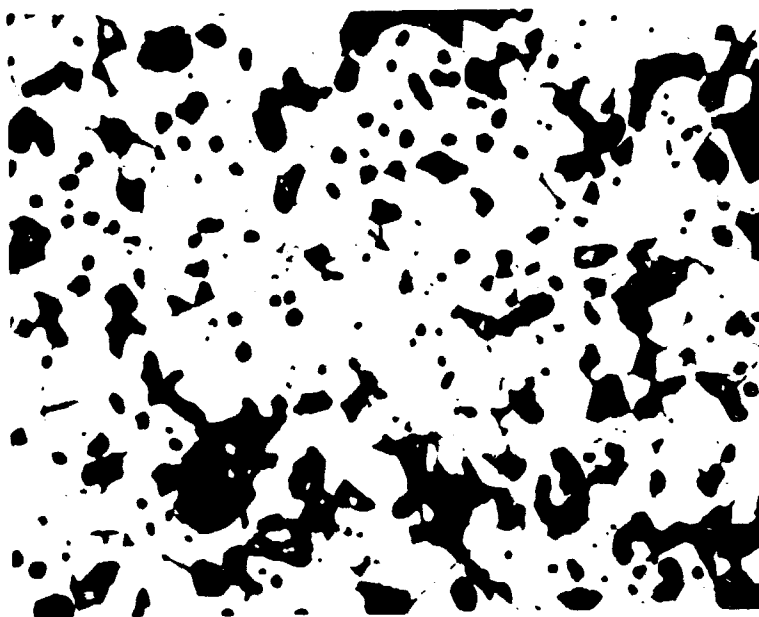
250X

**FIGURE 8.** Polished and Etched Specimens of Hot Pressed ( $>99.9\%$ ) Aluminum Oxide Shown at Three Different Magnifications



**FIGURE 9. Polished and Etched Sample of Lucalox at 250X and 500X**

**WAED64. 75E-29**



**FIGURE 10. Micrograph at 250X of a Polished and Etched Specimen of Triangle RR (+99.7% Recrystallized Alumina, Morganite, Inc.)**

are evident within the grains (rounded black spots).

Grain pull-outs were a problem with this specimen and these are evident by the larger black areas. Neither the Lucalox nor hot pressed aluminum oxide materials show evidence of pores in their microstructures, indicating very little open or closed porosity is present.

b. Resistivity Measurements

Several hot pressed disks were electroded (fired on platinum) using the techniques outlined in the Second Quarterly Progress Report. In addition, the same fixturing and furnace was used as previously described. D-C resistance measurements were made in flowing argon at various temperatures up to 850 C. Volume resistivity was calculated from sample and electrode dimensions. Resistivity vs. temperature is shown in Figure 11. The sensitivity of the test equipment available and high-sample resistivity did not allow resistance measurements to be made below 400 C. Nevertheless, the data obtained at higher temperatures show that the samples have resistivities in the same decade range as Lucalox and high purity, single crystal, synthetic sapphire. (Reference: Second Quarterly Report and Linde Co. Bulletin F-814-C.)

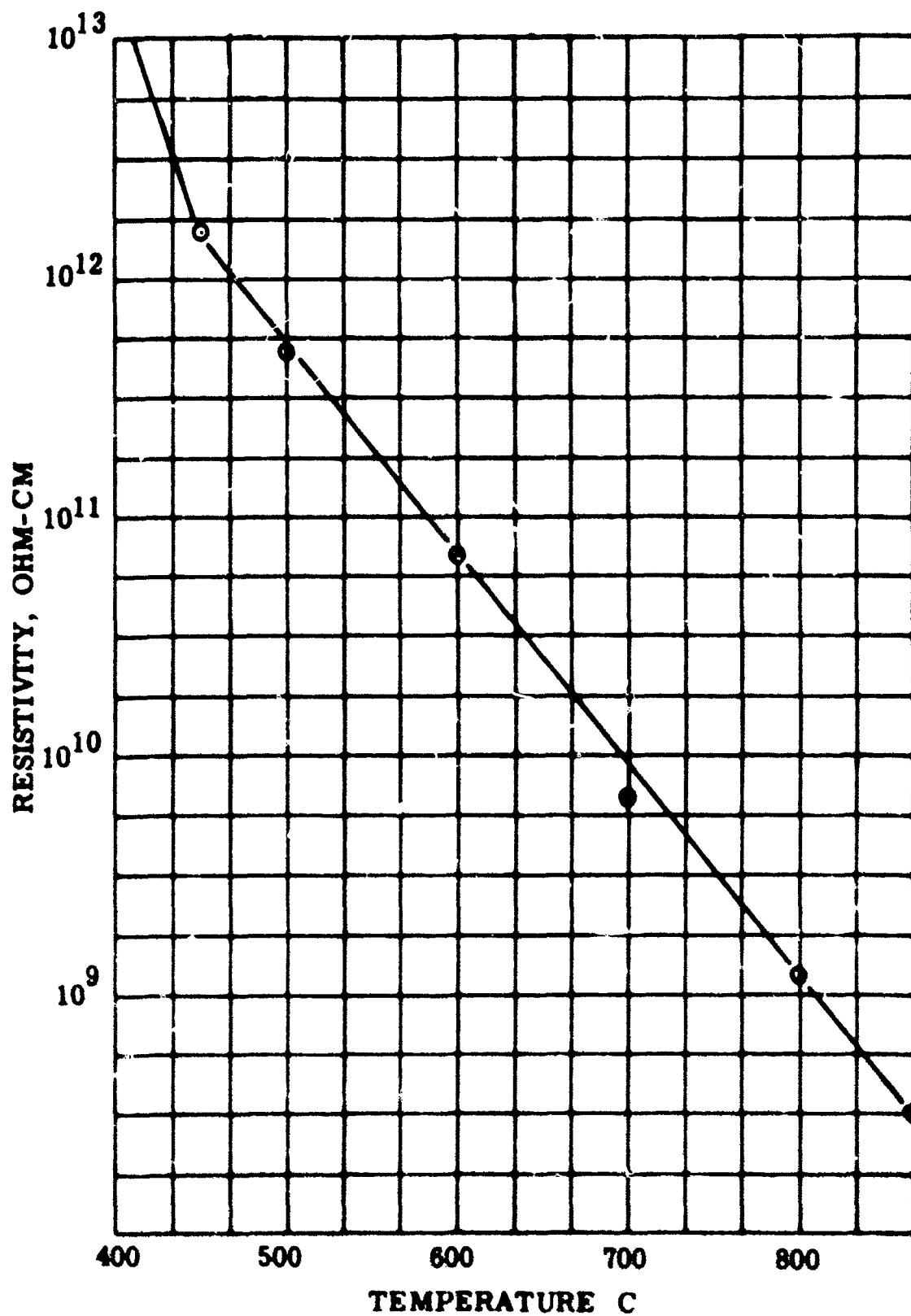


FIGURE 11. Resistivity Vs. Temperature For Hot Pressed Aluminum Oxide ( $>99.9\%$  Starting Material)

## C. Magnetic Properties

### 1. Comparison of Magnetic Properties - 5% Al-Fe, 3% Al-1%

#### Y-Fe and Cubex (Cube oriented 3 1/4% Si-Fe) Alloys

The total a-c core loss, apparent power, and d-c magnetization properties of 5% Al-Fe and 3% Al, 1% Y-Fe alloys are shown in Figures 12 through 23. The 5% Al-Fe alloy was annealed at 980 C in hydrogen both dry (entrance dewpoint, -60 C) and wet (bubbled through water before entering furnace). The oxide developed using wet hydrogen on the 5% Al-Fe spalled off as a fine powder showing a complete lack of adherence.

The 3% Al, 1% Y-Fe alloy was annealed at 1180 C in hydrogen both dry (entrance dewpoint, -60 C) and wet (bubbled through water before entering furnace). The oxide developed using wet hydrogen was thin and tightly adhering. The annealing temperature at 1180 C, was used to determine whether further grain growth would occur over the lower annealing temperature used for the 5% Al-Fe alloy of 980 C. No further grain growth occurred.

The comparative magnetic properties of 5% Al-Fe, 3% Al-Fe, 1% Y-Fe alloys, and Cubex, all 0.011 inch thick, are shown in Table III. The Al-Fe alloys show no improvement in either a-c magnetization at 400 cps or a-c core loss properties over Cubex.

2. Potassium Vapor Exposure - Effect on Magnetic Properties

The effect of exposure to potassium vapor at 600 C for 500 hours on Cubex, Hiperco 27, and Armco Ingot Iron is shown in Table IV. There is no significant effect on a-c core loss and d-c magnetization at the proposed operating level of 80 KL/in.<sup>2</sup>. The complete a-c core loss, apparent power, and d-c magnetization properties of Cubex, Hiperco 27, and Armco Ingot Iron are shown in Figures 24 through 32.

3. Chemical Analysis of Magnetic Materials

In Table V, the chemical analysis of the experimental heats of 5% Al-Fe and 3% Al, 1% Y-Fe are shown. The chemistry goals were reached except on Heat 664 where the yttrium is lower than desired.

An analysis made of a sample of Cubex after exposure to potassium vapor at 600 C for 500 hours showed no significant change in silicon content from normal. There was 2.9% silicon in the sample after exposure.

TABLE III. Comparative Magnetic Properties of Cubex: 5% Al-Fe; 3% Al, 1% Y-Fe

Material	Heat Treatment	Insulation	A-C Properties P <sub>c</sub> , Total Core Loss, 2 in watts/lb. @ 80 KI./in. 2 (~12.4 KG) @ 400 cps	D-C Properties H, Oersted, 2 @ B-80 KI./in. 2 (~12.4 KG)
0.011 inch Cubex	900 C Dry H <sub>2</sub>	Mica Aluminum Ortho Phosphate	13	1.8
0.011 inch-5% Al-Fe Core 7	980 C Wet H <sub>2</sub>	0.25 Mil Mylar	16.5	14
0.011 inch-5% Al-Fe Core 6	980 C Dry H <sub>2</sub>	0.25 Mil Mylar	16.6	10.2
0.011 inch-3% Al, 0.63% Y-Fe Core 9A	1180 C Wet H <sub>2</sub>	Oxide	30.5	14
0.011 inch-3% Al, 0.88% Y-Fe Core 8	1180 C Dry H <sub>2</sub>	0.25 Mil Mylar	37	18.8

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TABLE IV. Comparative Magnetic Properties of Cubex, Hiperco 27, and Armco Ingot Iron Before and After Potassium Vapor Exposure

Material	Insulation	Initial Heat Treatment	Special Treatment	A-C Properties	D-C Properties
				$P_c$ , Total Core Loss in Watts/lb. @ B-80 KI./in. 2 (~12.4 KG) @ 400 cps	$H_c$ , Oersted, 2 @ B-80 KI./in. 2 (~12.4 KG)
0.006 inch Cubex, Core 2	0.25 Mil Mylar	Stress Relief-Annealed, 900 C, 1.5 Hour, H <sub>2</sub>	None	7.8	3.6
0.006 inch Cubex, Core 2	0.25 Mil Mylar	Stress Relief-Annealed, 900 C, 1.5 Hour, H <sub>2</sub>	Exposed to K Vapor @ 600 C, 500 Hours	8.0	3.8
0.008 inch Hiperco 27, Core 1A	Linde A	Annealed, 900 C, 1 Hour, H <sub>2</sub>	None	21.3	-
0.008 inch Hiperco 27, Core 1A	Linde A	Annealed, 900 C, 1 Hour, H <sub>2</sub>	Exposed to K Vapor @ 600 C, 500 Hours	20.8	6.8
0.008 inch Armco Ingot Iron, Core 3	0.25 Mil Mylar	Annealed, 900 C, 1.5 Hour, H <sub>2</sub>	None	23.0	4.4
0.008 inch Armco Ingot Iron, Core 3	0.25 Mil Mylar	Annealed, 900 C, 1.5 Hour, H <sub>2</sub>	Exposed to K Vapor @ 600 C, 500 Hours	22.2	4.0

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TABLE V. Chemical Analysis - Percent by Weight of Iron Base Alloys

<u>Elements</u>	<u>5% Al Heats</u>		<u>3% Al, 1% Y Heats</u>	
	<u>662</u>	<u>663</u>	<u>664</u>	<u>665</u>
Total C	0.02	0.02	0.03	0.03
Mn	< 0.01	< 0.01	< 0.01	< 0.01
P	0.002	0.002	0.003	0.003
S	0.008	0.006	0.006	0.006
Al	4.92	4.89	2.93	2.90
Y	< 0.01	< 0.01	0.62	0.88

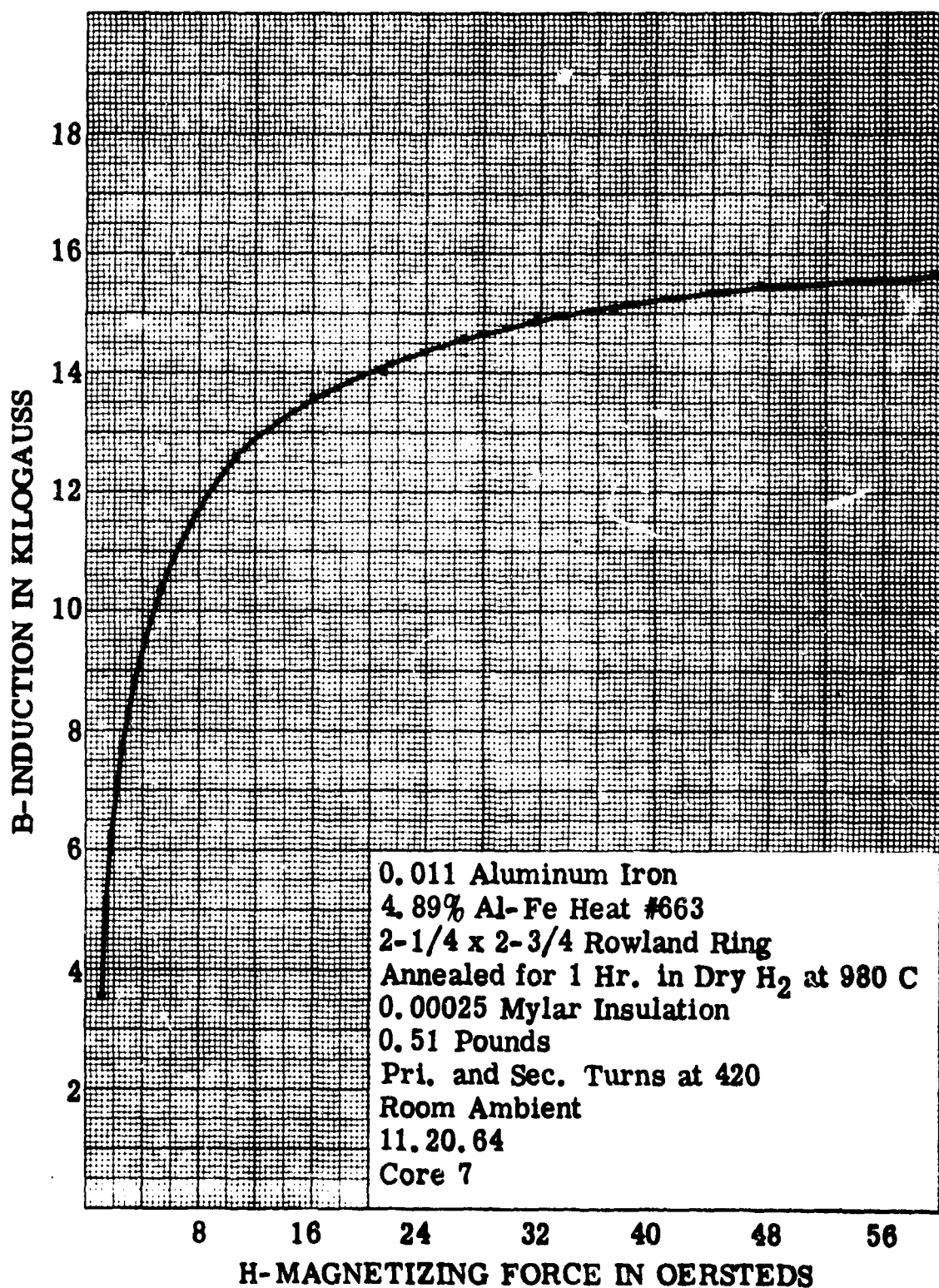
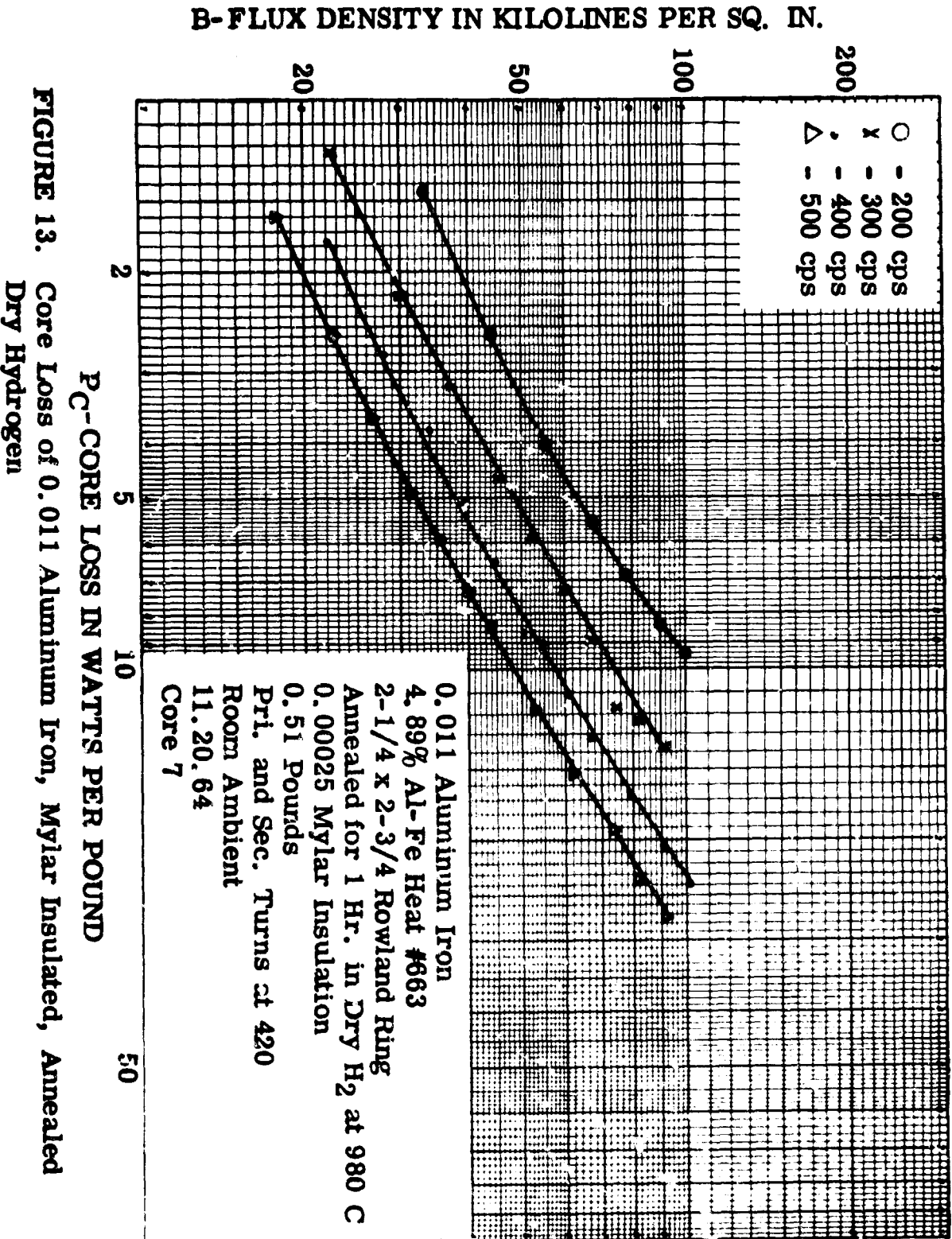


FIGURE 12. D-C Magnetization of 0.011 Aluminum Iron, Mylar Insulated, Annealed Dry Hydrogen



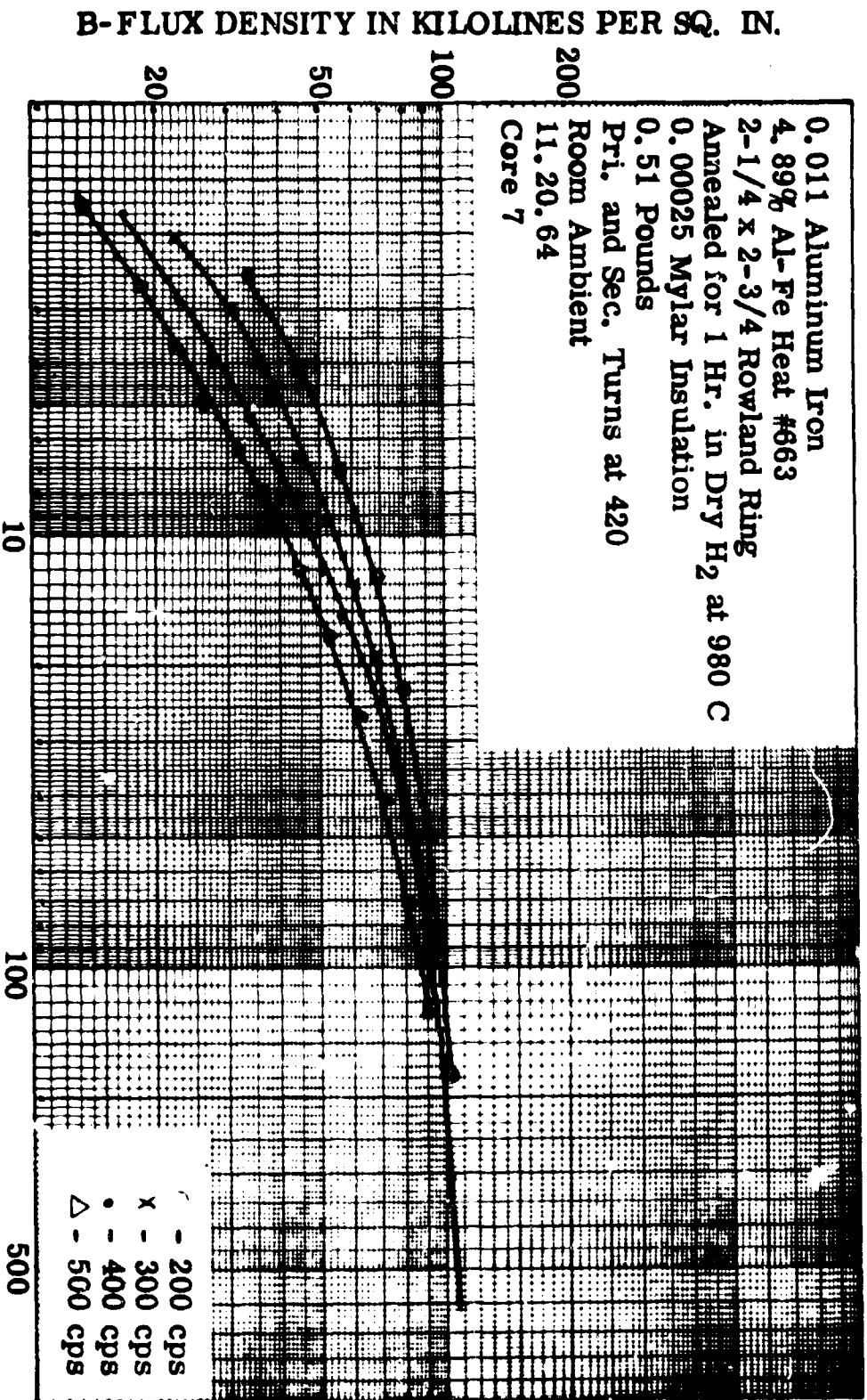


FIGURE 14. Apparent Core Loss of 0.011 Aluminum Iron, Mylar Insulated, Annealed Dry Hydrogen

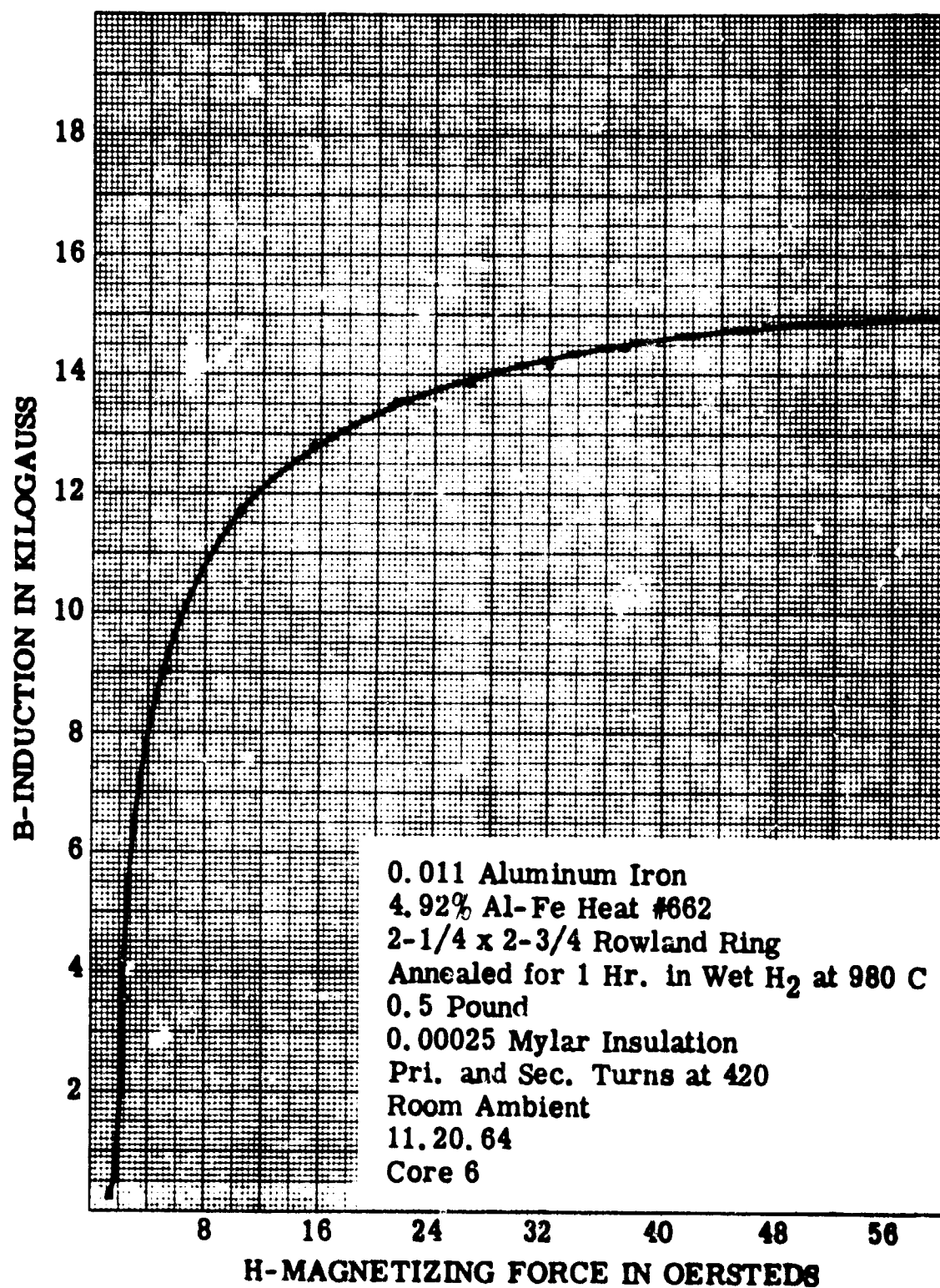
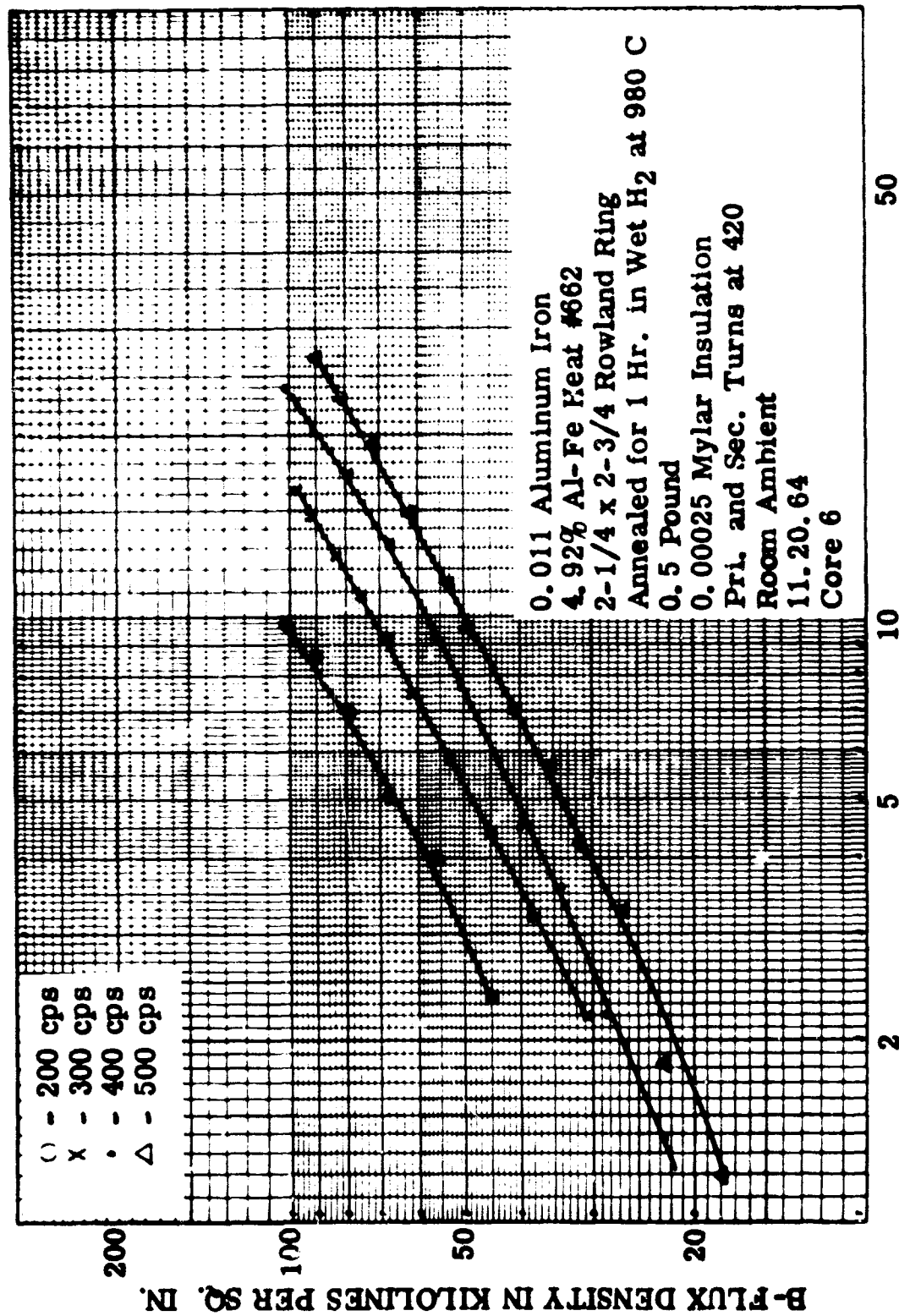
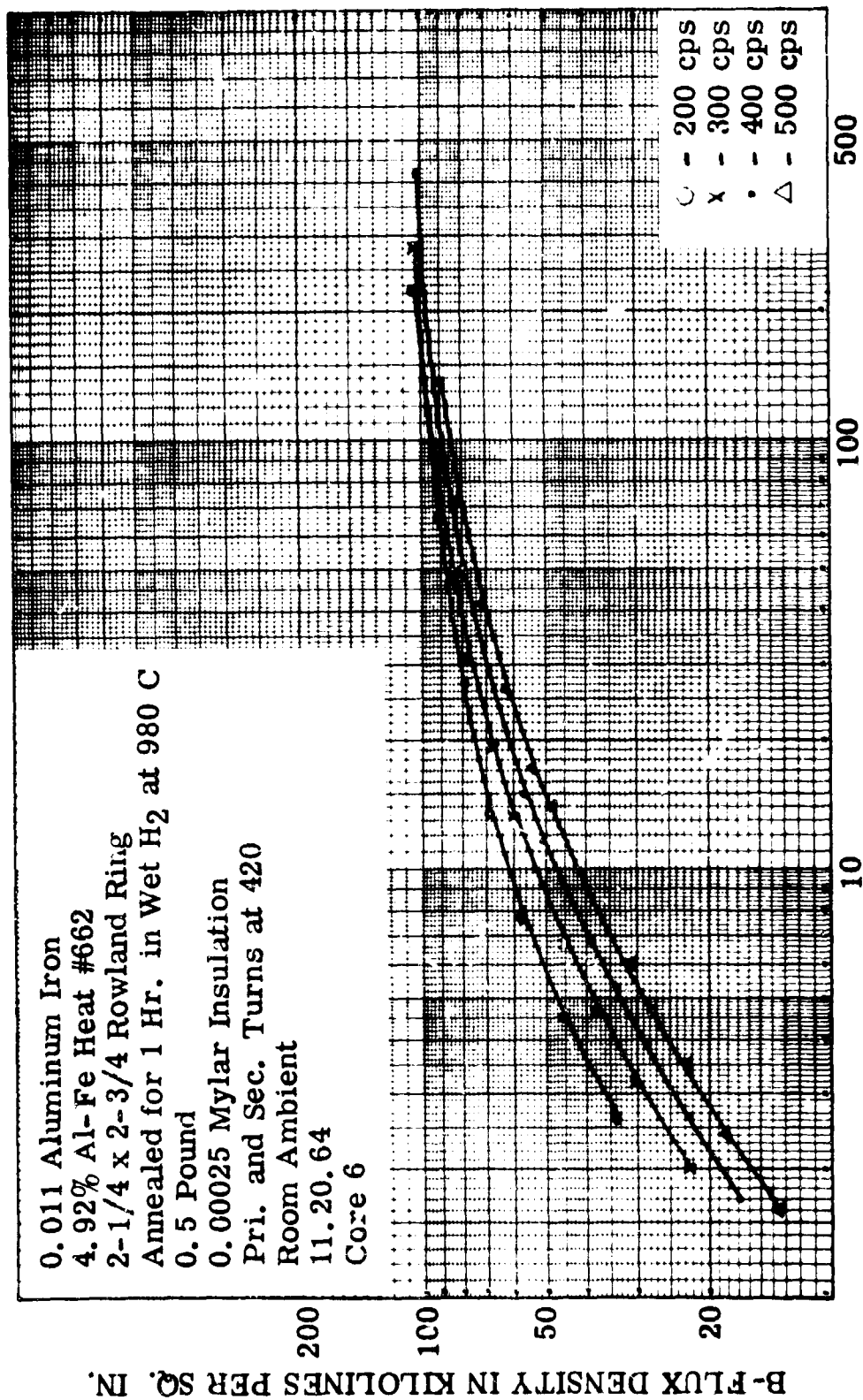


FIGURE 15. D-C Magnetization of 0.011 Aluminum Iron, Mylar Insulation, Annealed Wet Hydrogen



**FIGURE 16.** Core Loss of 0.011 Aluminum Iron, Mylar Insulated, Annealed Wet Hydrogen



PA-APPARENT CORE LOSS IN VOLT-AMPS PER POUND

FIGURE 17. Apparent Core Loss of 0.011 Aluminum Iron, Mylar Insulated, Annealed Wet Hydrogen

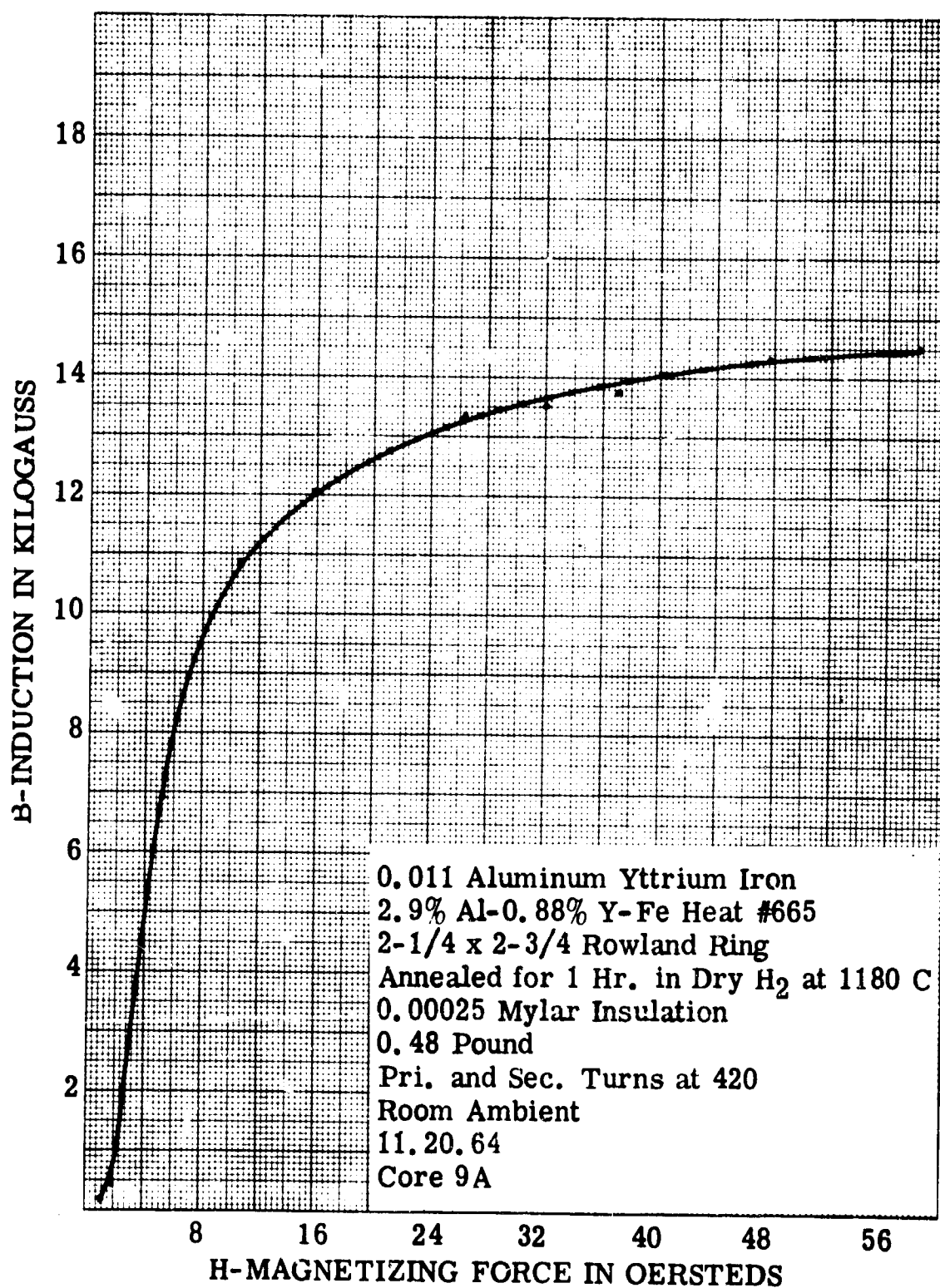


FIGURE 18. D-C Magnetization of 0.011 Aluminum Yttrium Iron, Mylar Insulated, Annealed Dry Hydrogen

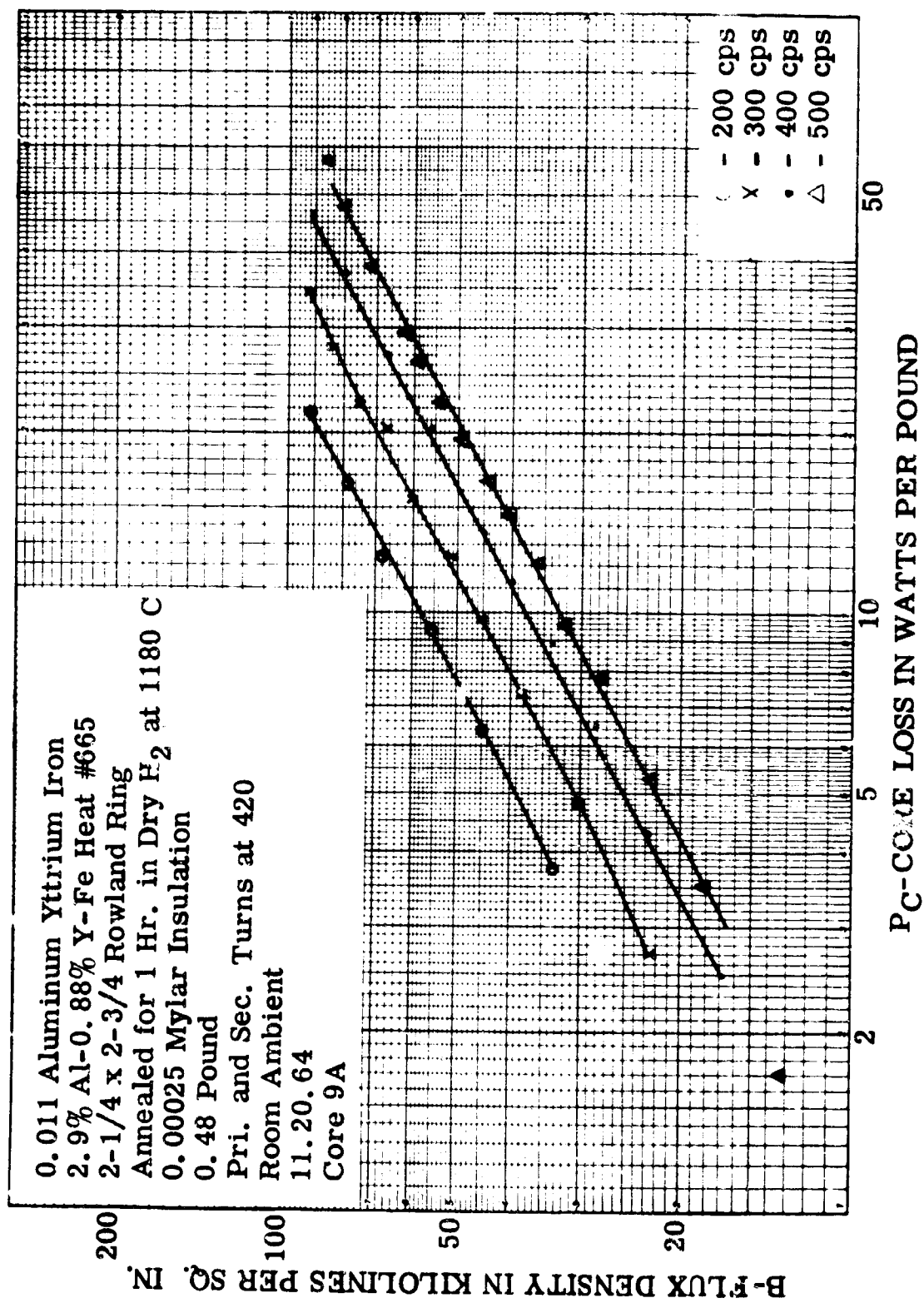
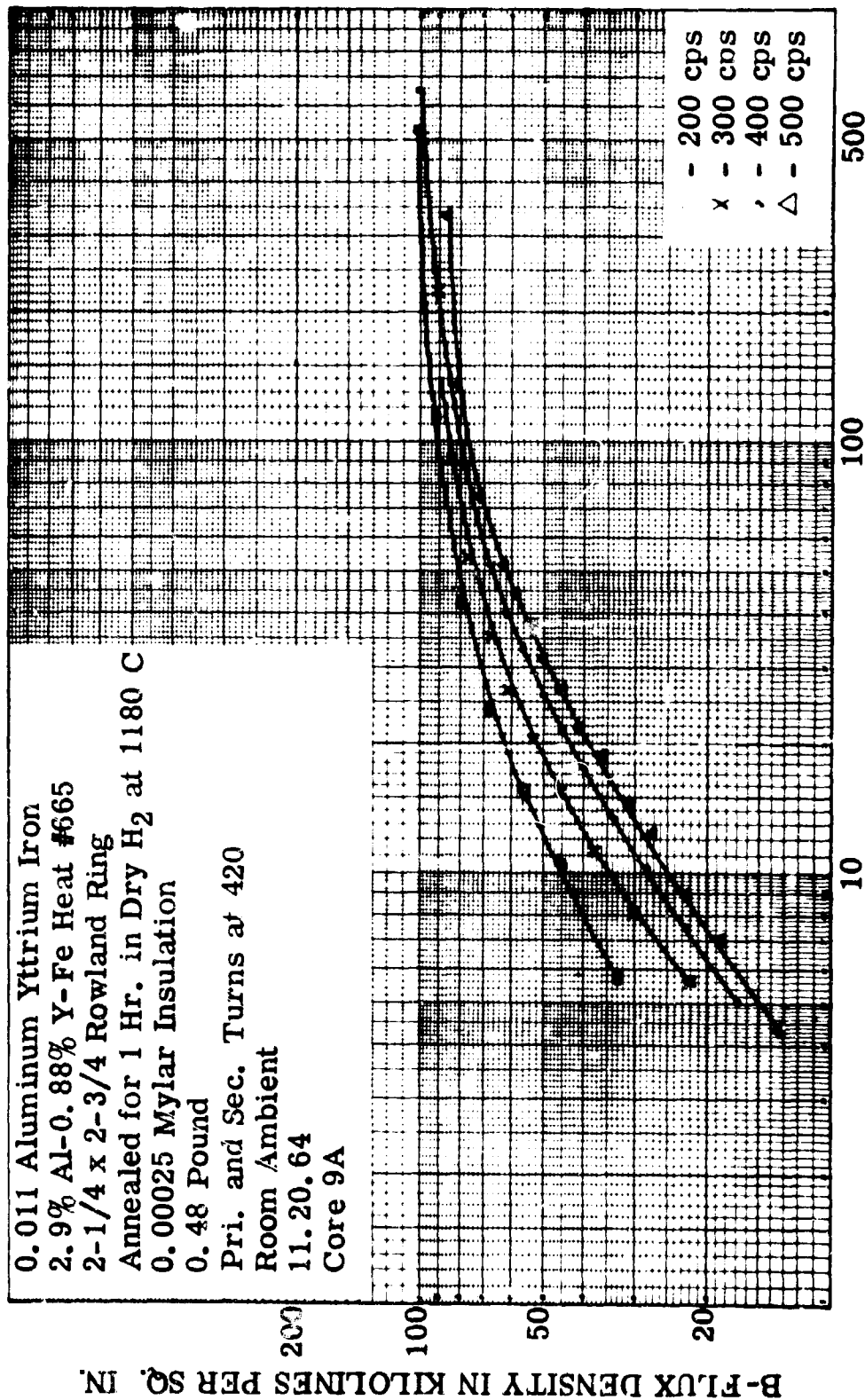


FIGURE 19. Core Loss of 0.011 Aluminum Yttrium Iron, Mylar Insulated, Annealed Dry Hydrogen



**FIGURE 20. Apparent Core Loss of 0.011 Aluminum Yttrium Iron, Mylar Insulated, Annealed Dry Hydrogen**

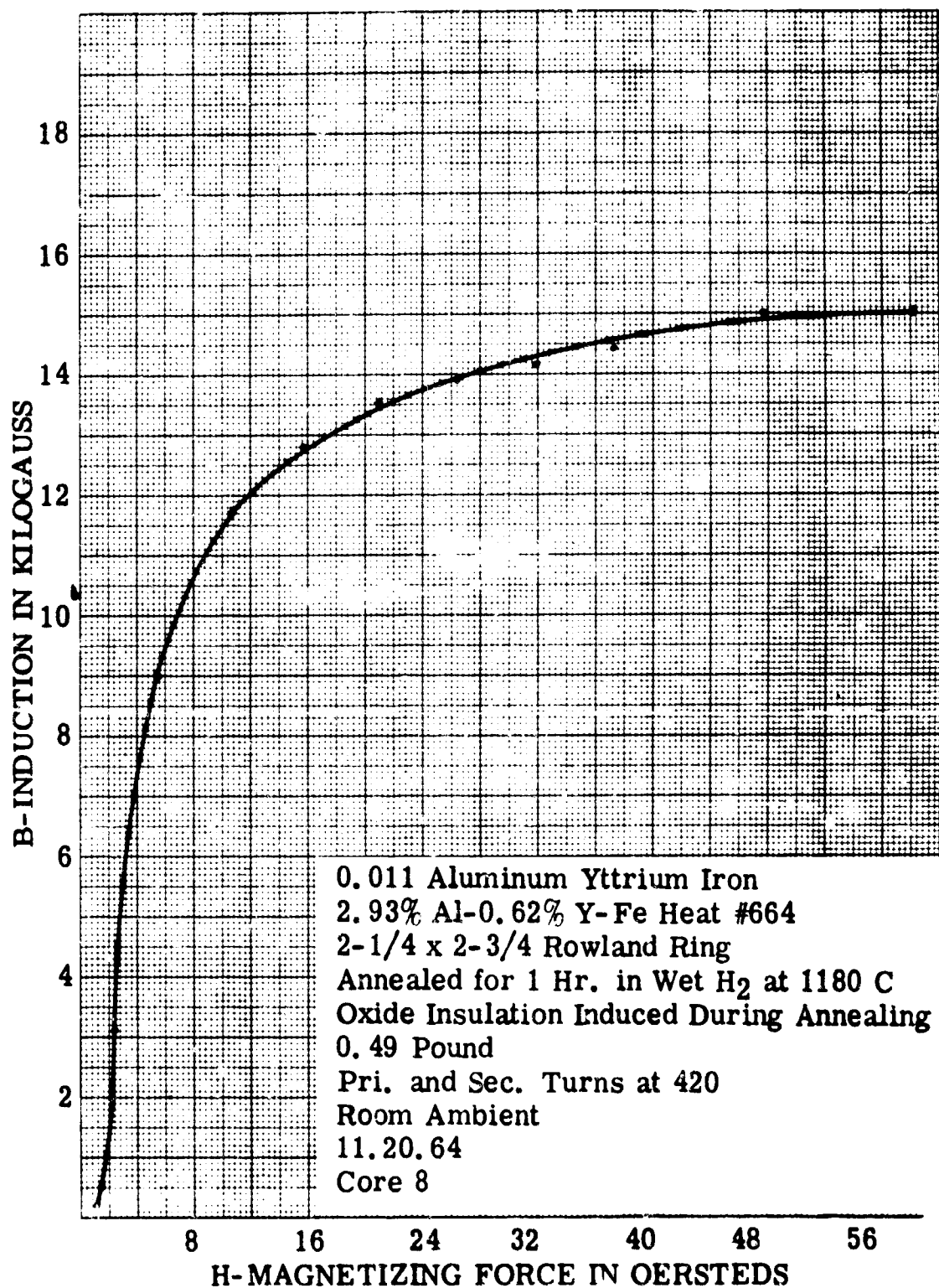


FIGURE 21. D-C Magnetization of 0.011 Aluminum Yttrium Iron, Mylar Insulated, Annealed Wet Hydrogen

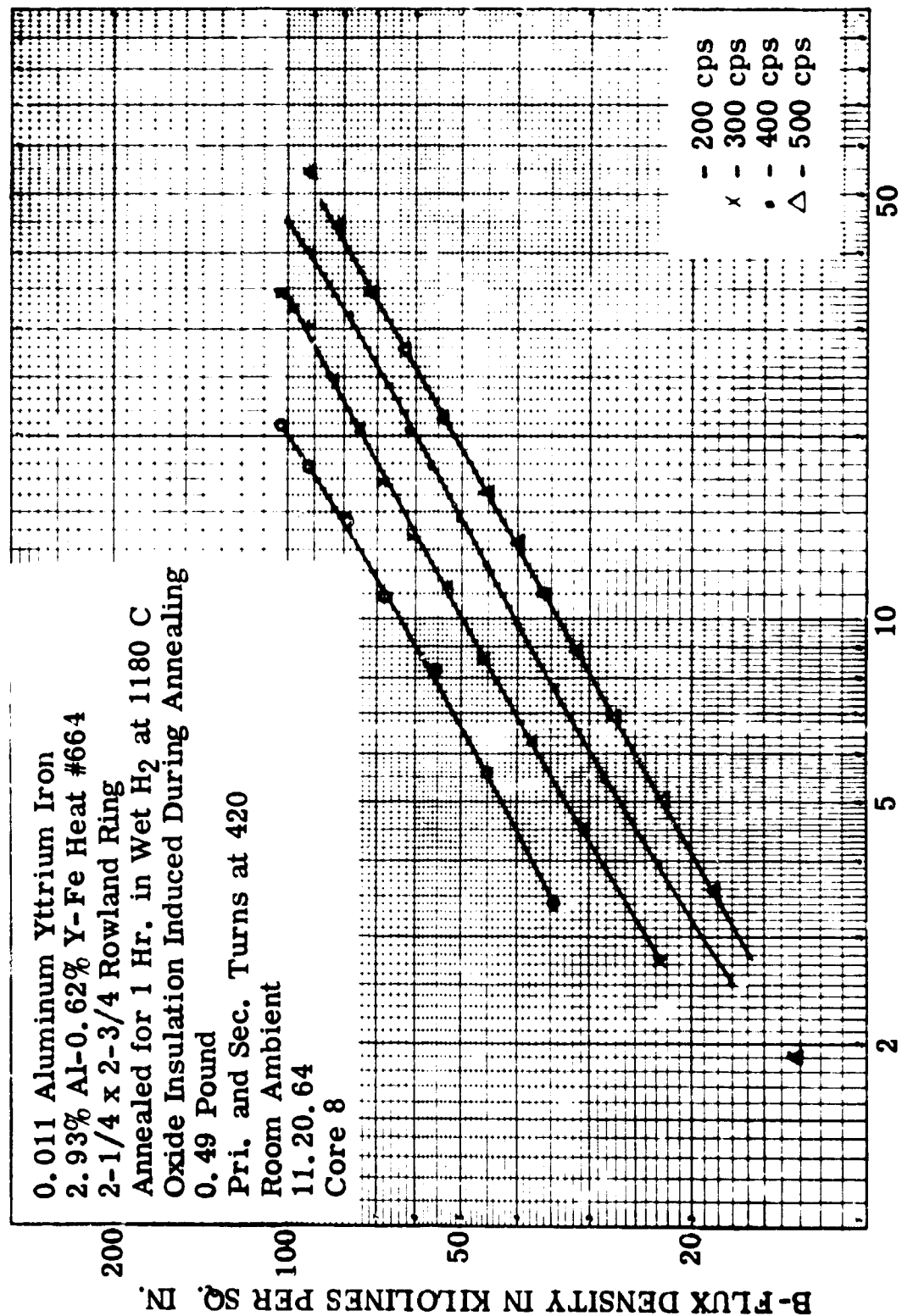


FIGURE 22. Core Loss of 0.011 Aluminum Yttrium Iron, Mylar Insulated, Annealed Wet Hydrogen

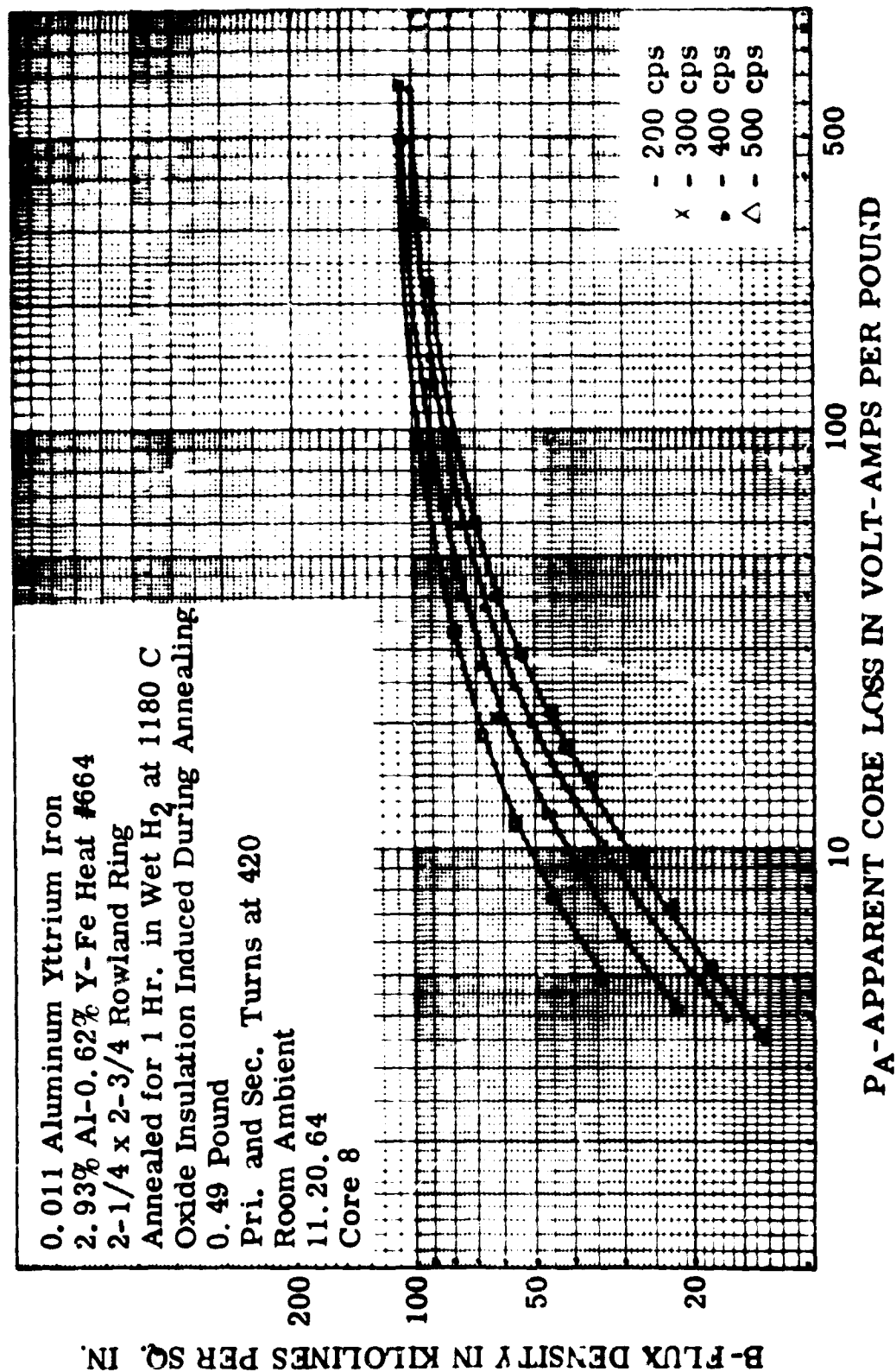


FIGURE 23. Apparent Core Loss of 0.011 Aluminum Yttrium Iron, Mylar Insulated, Annealed Wet Hydrogen

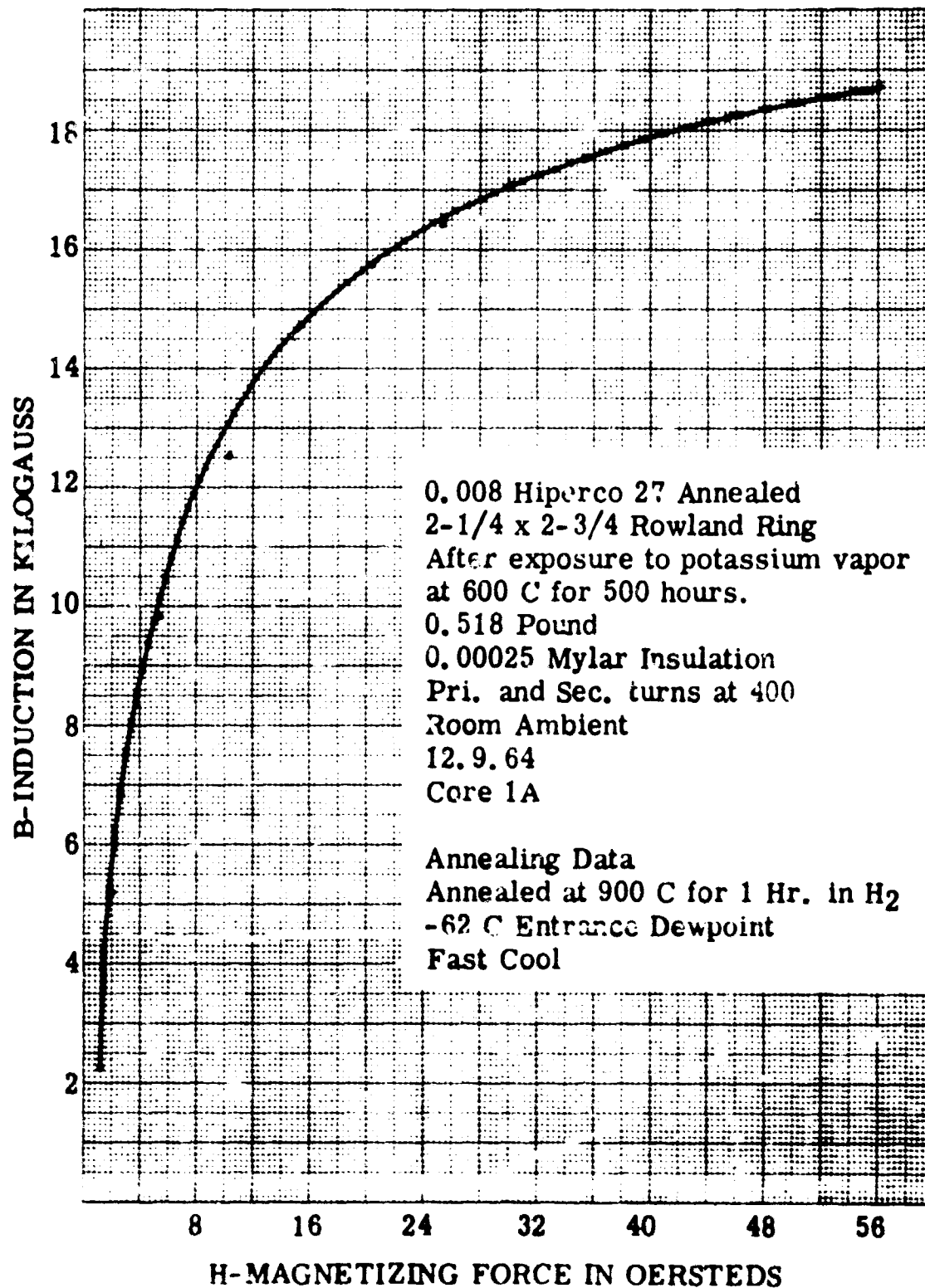


FIGURE 24. D-C Magnetization of 0.008 Hiperco 27, Mylar Insulated, After Exposure to Potassium Vapor

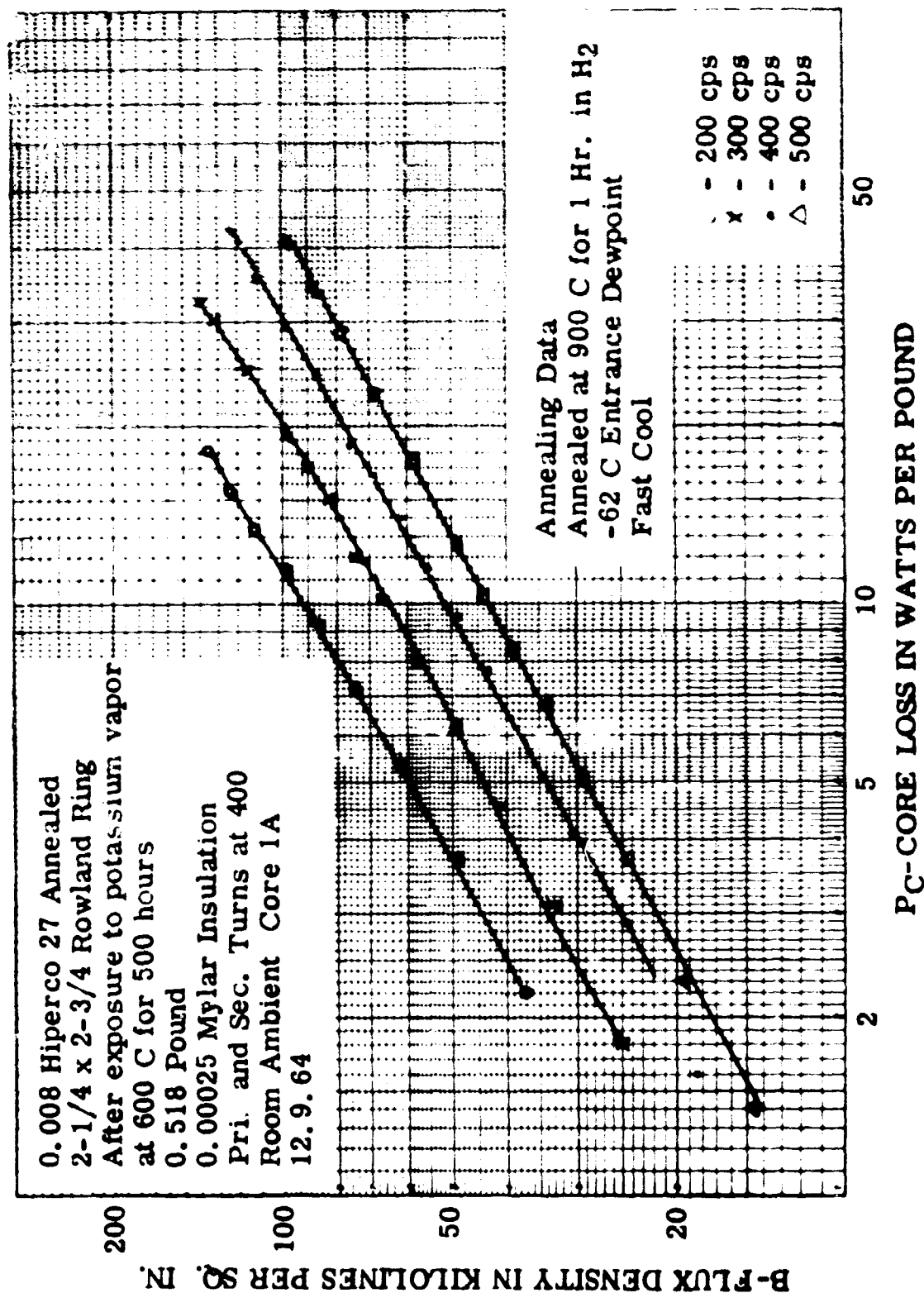


FIGURE 25. Core Loss of 0.008 Hiperco 27, Mylar Insulated,  
After Exposure to Potassium Vapor

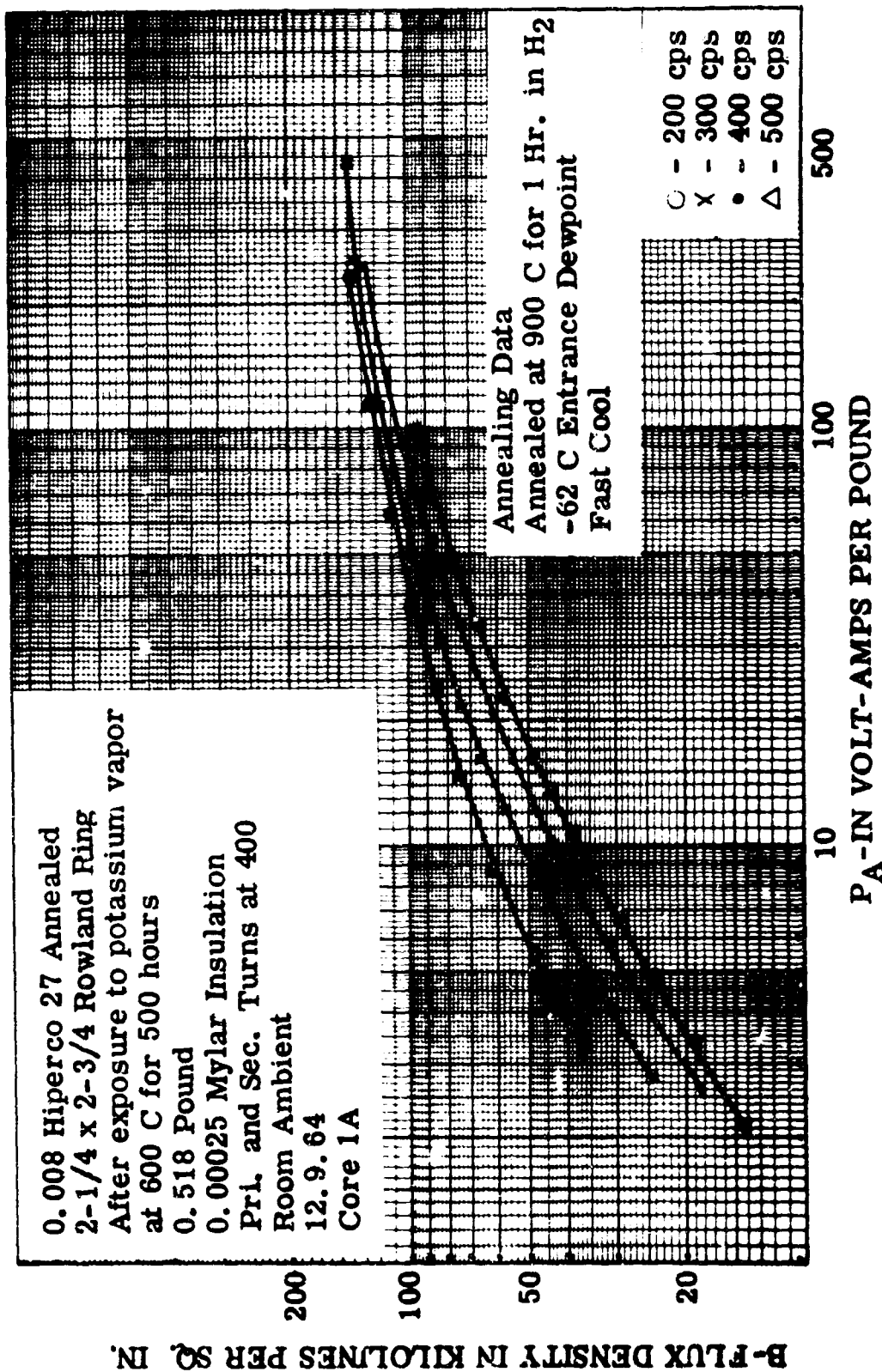


FIGURE 26. Apparent Core Loss of 0.008 Hiparco 27, Mylar Insulated,  
After Exposure to Potassium Vapor

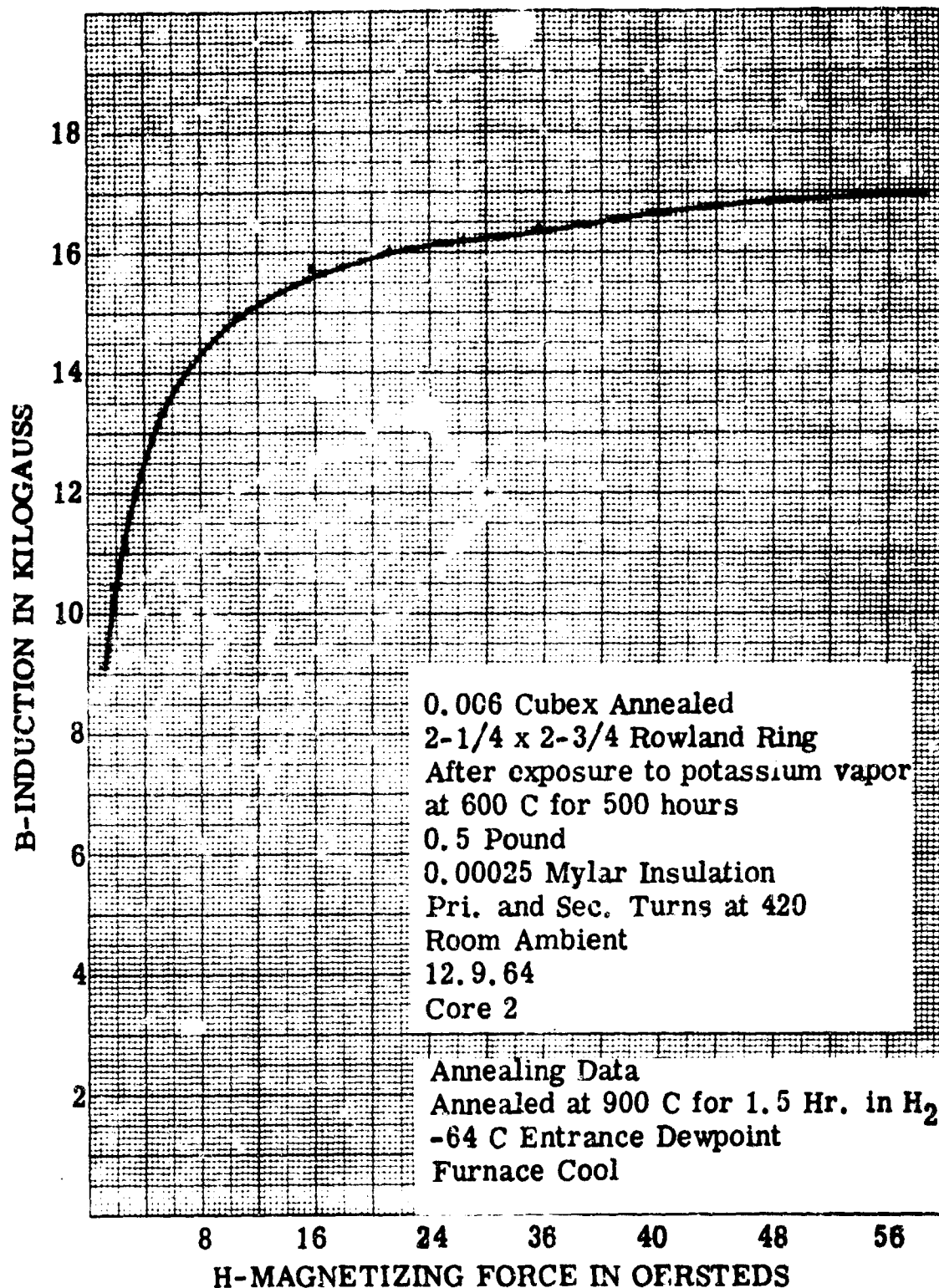
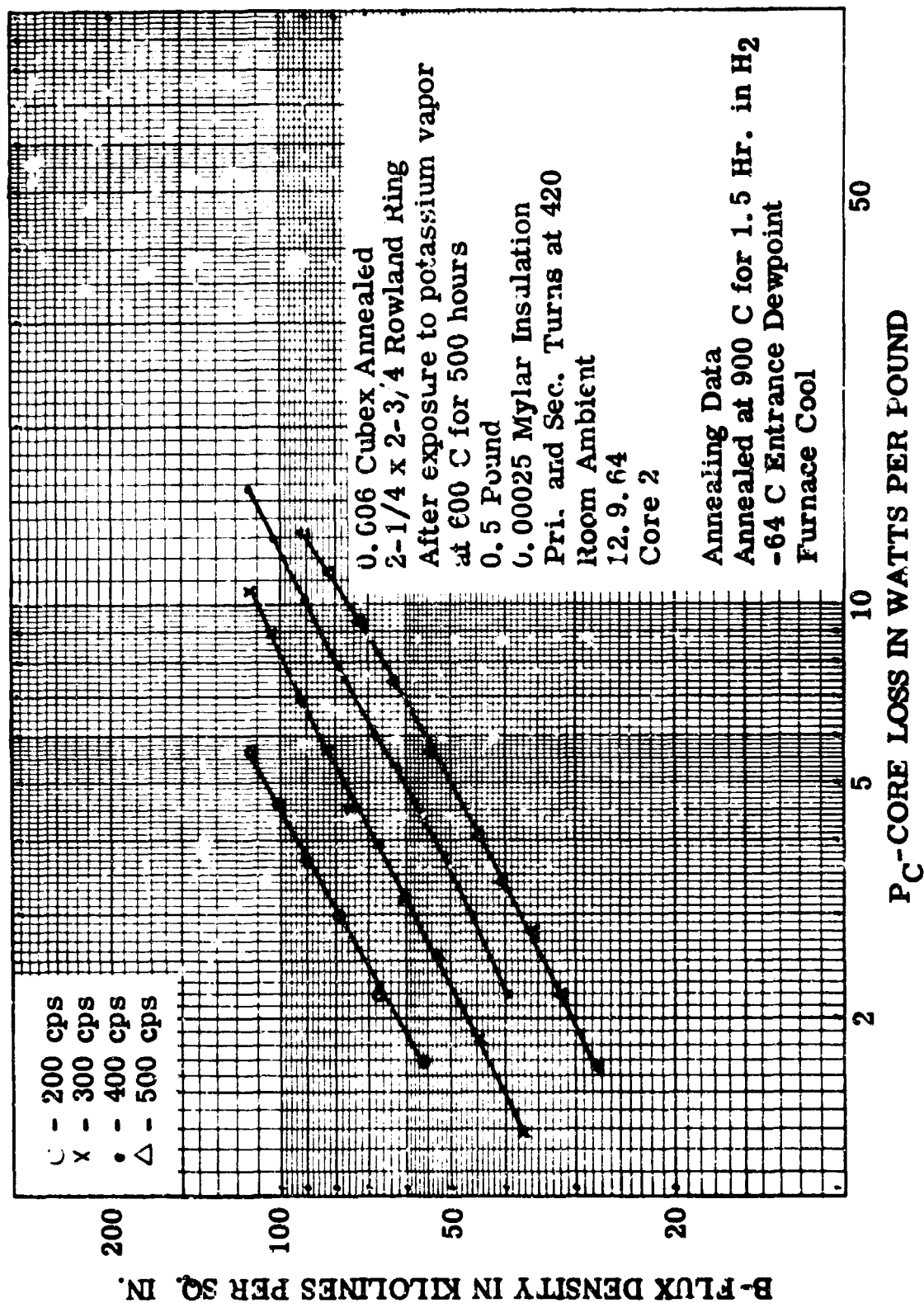
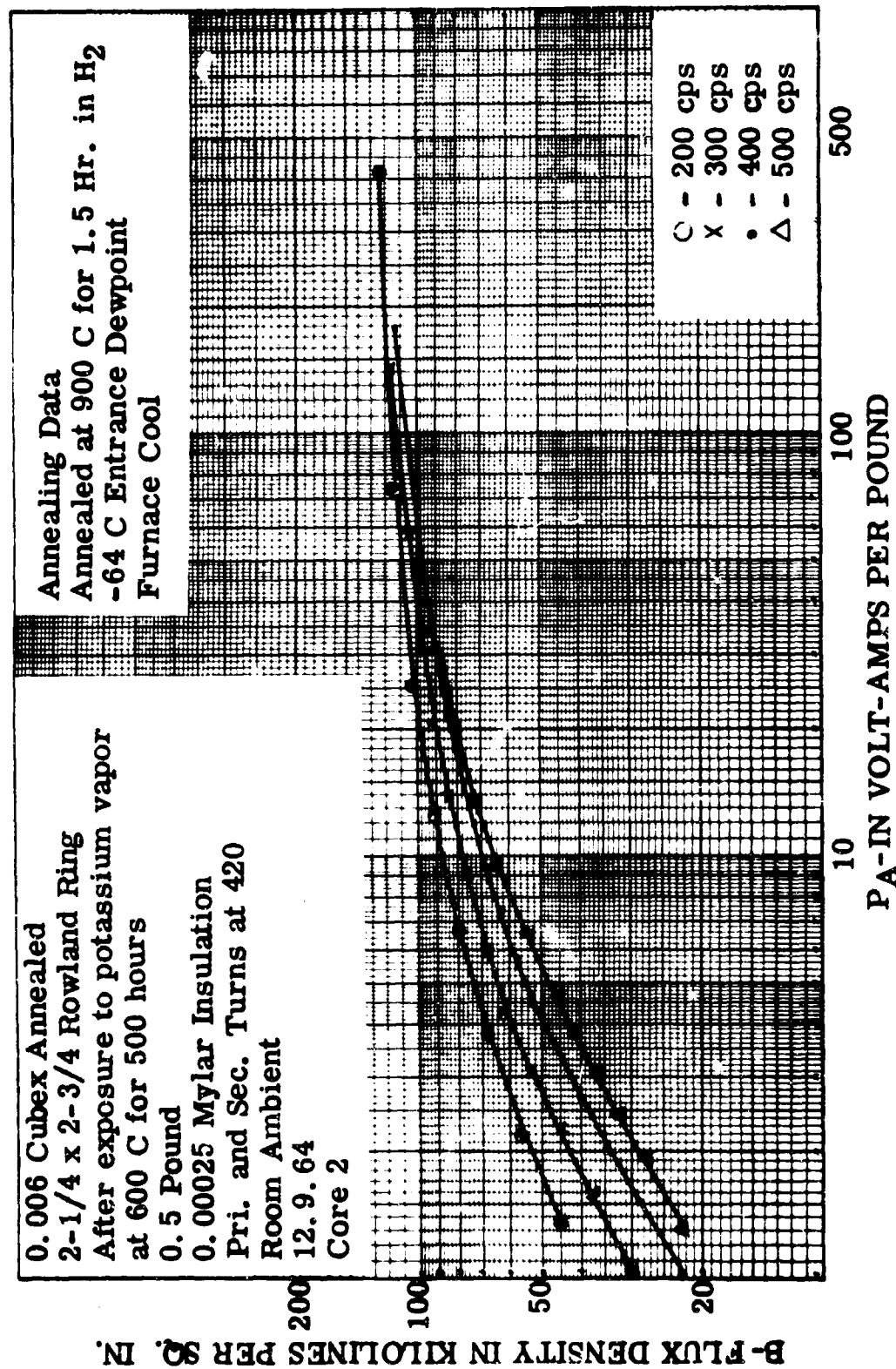


FIGURE 27. D-C Magnetization of 0.006 Cubex, Mylar Insulated, After Exposure to Potassium Vapor



**FIGURE 28. Core Loss of 0.006 Cubex, Mylar Insulated, After Exposure to Potassium Vapor**



**FIGURE 29. Apparent Core Loss of 0.006 Cubex, Mylar Insulated,  
 After Exposure to Potassium Vapor**

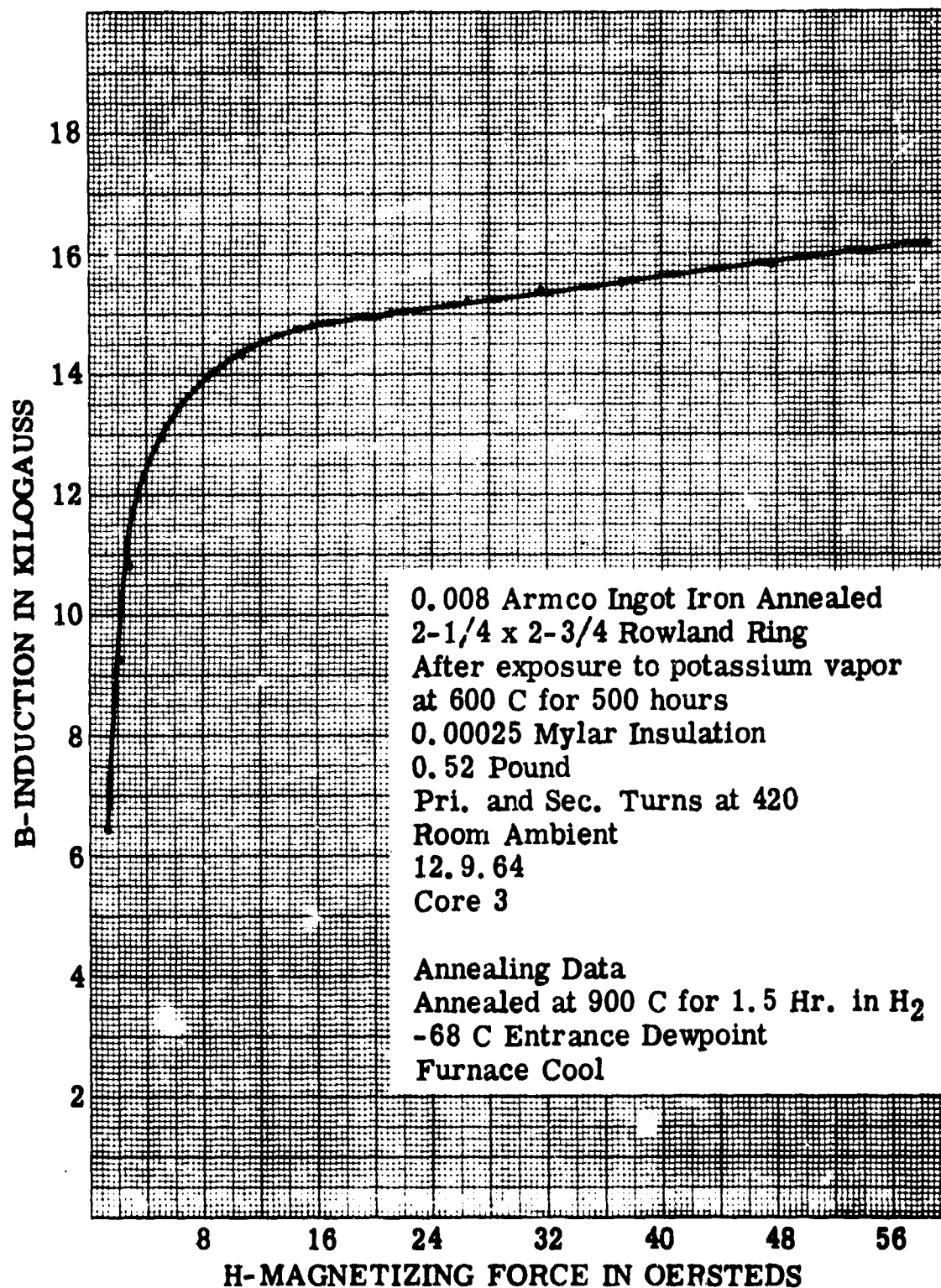


FIGURE 30. Magnetization of 0.008 Armco Ingot Iron, Mylar Insulated, After Exposure to Potassium Vapor

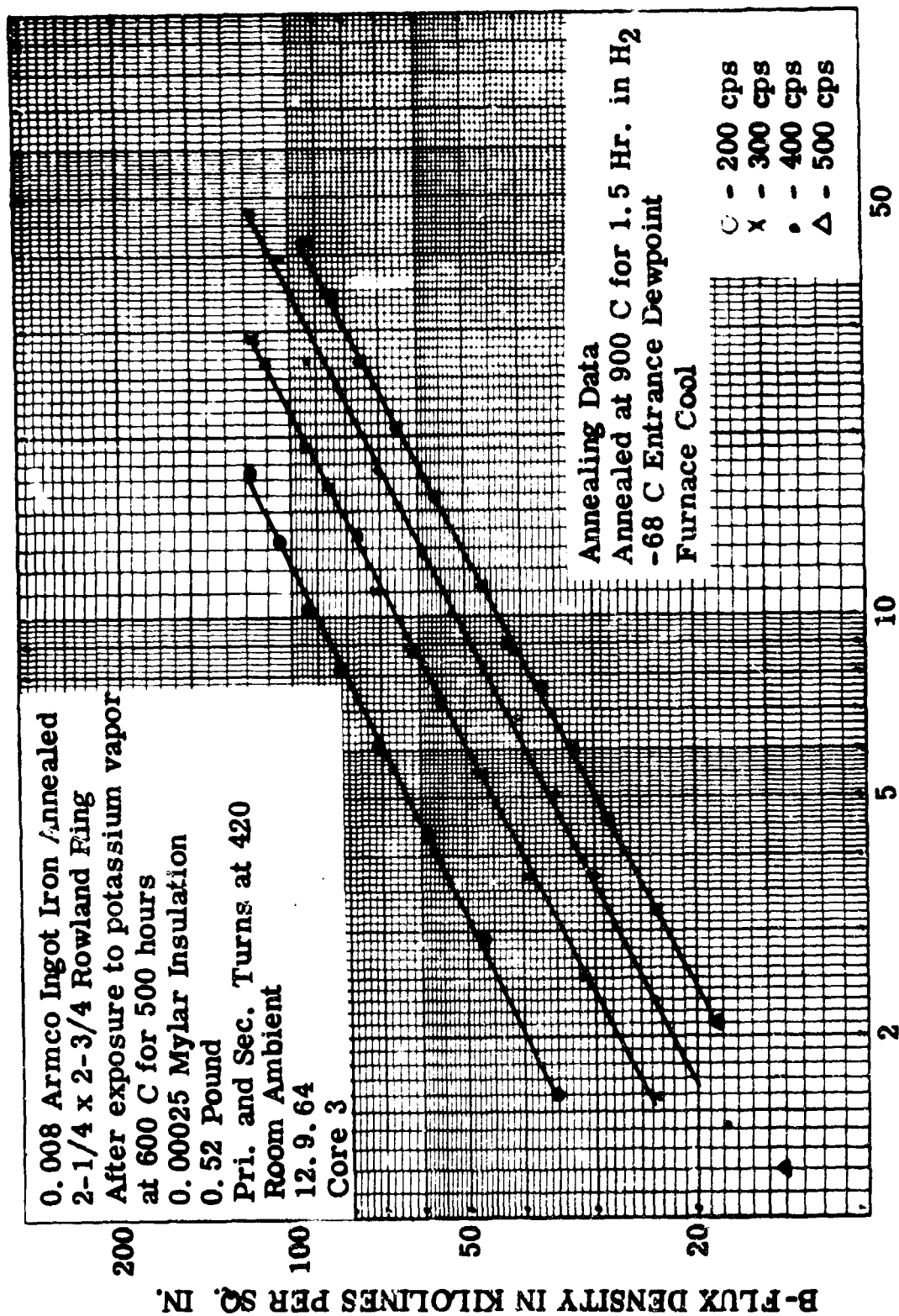


FIGURE 31. Core Loss of 0.008 Armco Ingot Iron, Mylar Insulated,  
After Exposure to Potassium Vapor

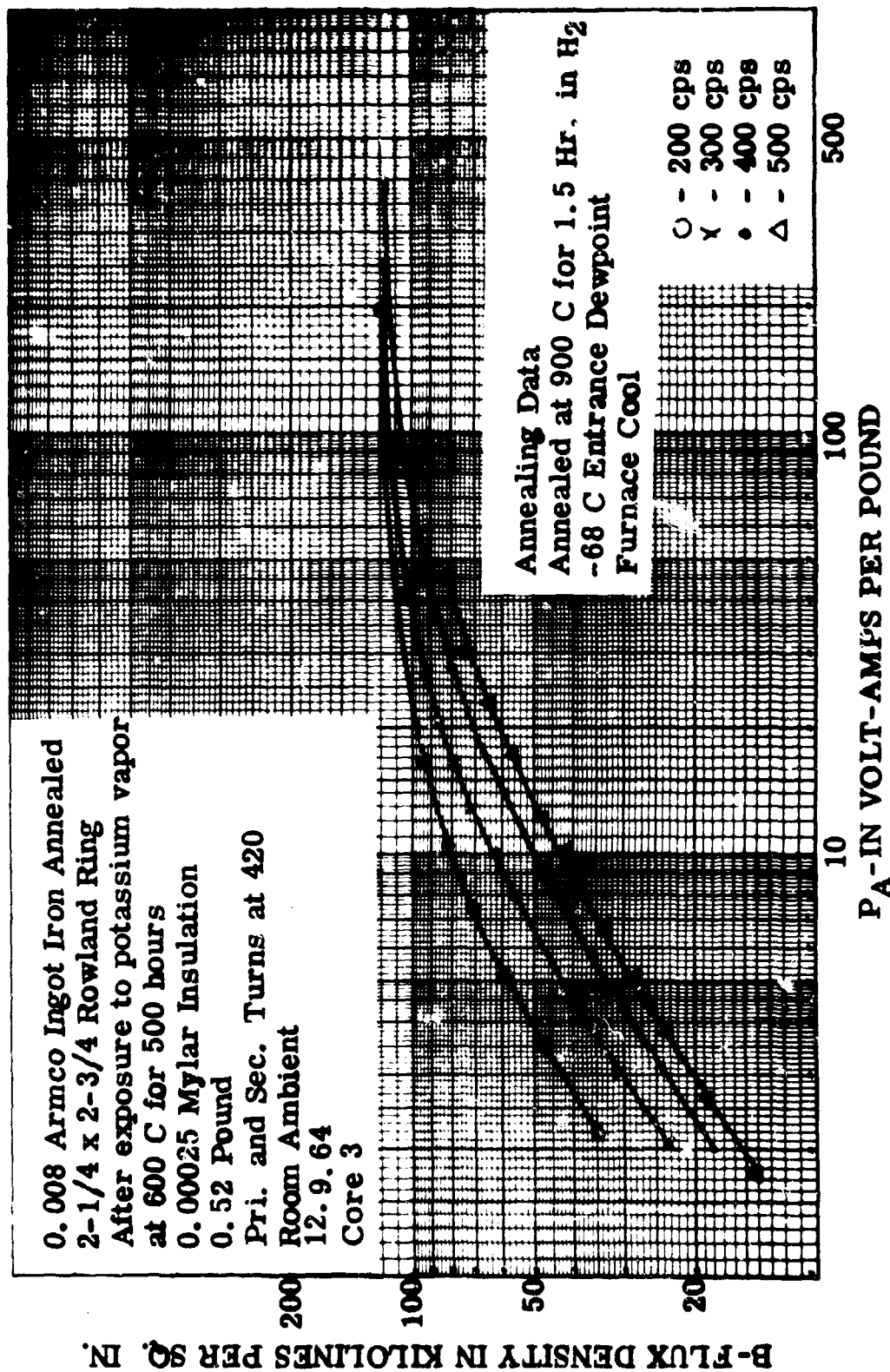


FIGURE 32. Apparent Core Loss of 0.008 Armco Ingot Iron, Mylar Insulated, After Exposure to Potassium Vapor

## D. Conductors

### 1. Nickel-Clad Silver

#### a. Electroplating Exposed Silver Core and Silver Braze Joint

Two samples of the (28%) nickel-clad silver conductor were prepared for nickel plating. One sample consisted of a 6.5 inch piece of No. 6 AWG nickel-clad silver conductor that had both ends rounded and polished. The other sample was two pieces of the same clad conductor with approximately a 0.75 inch length from one end of each piece ground through the nickel cladding to the silver. The exposed silver cores on the two pieces of conductor were clamped together side by side and silver brazed. The rough edges of this braze joint were rounded and polished before plating.

The nickel plating bath was a small laboratory set-up using a pyrex dish as a tank and a small laboratory stirrer to keep the solution agitated. The solution consisted of 60 oz/gal. nickel sulfamate, nickel metal 10.2 oz/gal., boric acid 4.0 oz/gal. and a commercial anti-pit agent (wetting agent) 0.05 oz/gal. The density of this solution varied from 29-31 degrees Baume'. The anodes were rolled and pre-polarized >99% purity nickel. The electrical conditions were 1-2.5 volts d-c at 0.25-0.5 amps. Temperature during plating varied from 32-60 C. A 0.005 inch-thick

coating of pore-free nickel was deposited over the exposed ends of the No. 6 AWG conductor in approximately six hours using this technique.

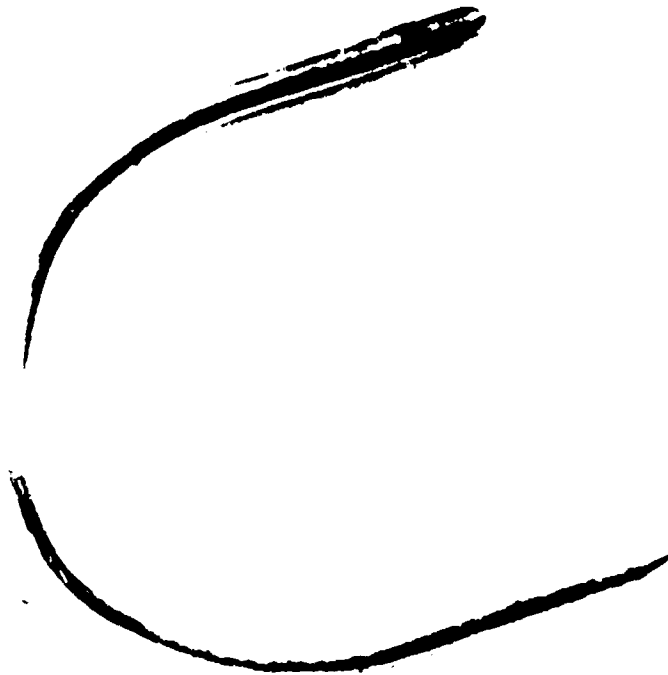
b. 500-Hour 600 C Potassium Vapor Exposure

Figure 33 shows a sample of No. 6 AWG conductor with the ends nickel plated. This sample has been exposed to potassium vapor at 600 C for 500 hours with no apparent damage to the conductor. A micrograph of a section made on the nickel-plated silver braze (BT alloy) joint that was also exposed to 600 C potassium vapor 500 hours, see Figure 34, shows this method of nickel-plating protects the silver from the corrosive action of potassium vapor.

2. Columbium-Clad Copper Conductor

a. Fabrication

The First Quarterly Report described several 70% columbium-clad conductors. The 70% columbium-clad copper and silver conductors were outside the requirement of 150% resistivity of OFHC copper. Columbium - 1% zirconium tubing 0.626 inches I. D. with a nominal wall thickness of 0.027 inches was obtained from Nuclear Metals Corporation, Summer-ville, Massachusetts. A dispersion-strengthened copper was obtained from Handy and Harman. This conductor material and tubing were drawn into wire by the Westing-house Metals Division at Blairsville, Pennsylvania. A



**FIGURE 33. Nickel-Clad Silver, No. 6 AWG, Conductor (28% Cladding) Exposed Ends Protected by Nickel Plating, After 500-Hour, 600 C Potassium Vapor Exposure**

**WAED64. 75E-61**

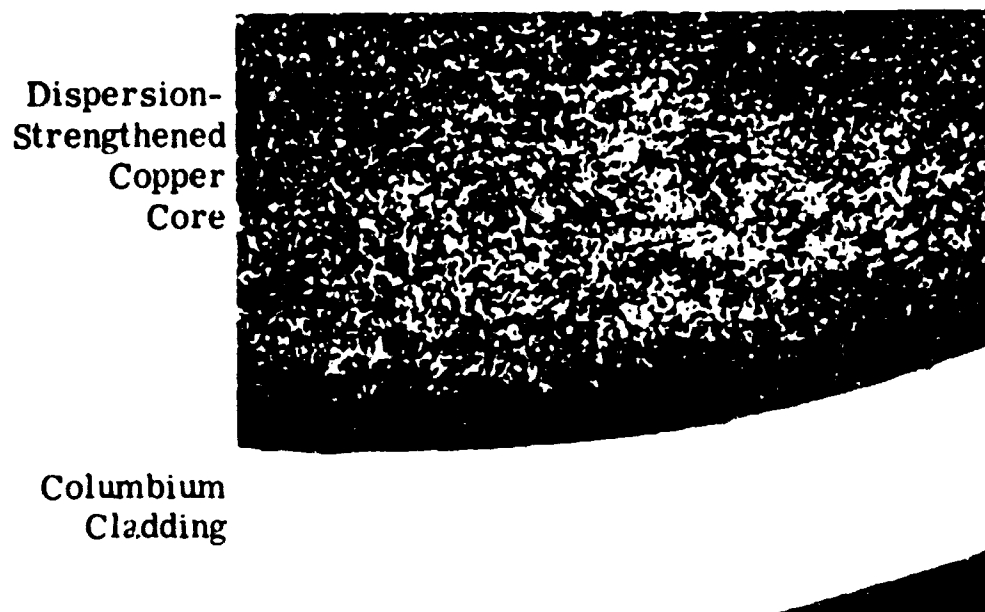
## Nickel Plating



Silver Core      Nickel Cladding      BT Silver Braze

**FIGURE 34.** Micrograph of Nickel Plated Silver Braze Joint  
After Exposure to 600 C Potassium Vapor for  
500 Hours

WAED64. 75E-62



**FIGURE 35.** (28%) Columbium - 1% Zirconium Clad Dispersion-Strengthened Copper Conductor, No. 6 AWG

No. 6 AWG (28%) columbium - 1% zirconium-clad dispersion-strengthened copper conductor was made by this technique. An edge of this clad conductor is shown in Figure 35.

b. Tensile Testing

A five inch length of this No. 6 conductor was clamped in the serrated jaws of a Baldwin Tensile Machine. The piece of conductor was pulled apart and the cladding came off the copper in the form of a small tube. The tensile strength of the conductor was approximately 62,000 psi at room temperature (23 C).

3. Rhodium Conductor - No. 22 AWG

a. Potassium Vapor Exposure

Two small three inch lengths of No. 22 AWG solid, float-zone-refined, rhodium conductor were obtained from the WPAFB project engineer, Mr. L. E. Schott, for 850 C potassium vapor exposure. One of these pieces was placed in a capsule with purified potassium and heated at 850 C for 172 hours. The weight change data in Table I shows a gain of 0.0011 grams, indicating the potassium vapor had very little corrosive action on the conductor. A sample was also vacuum annealed four hours at 850 C.

b. Physical Examination of Rhodium Conductor

Comparative hardness measurements on rhodium wire were

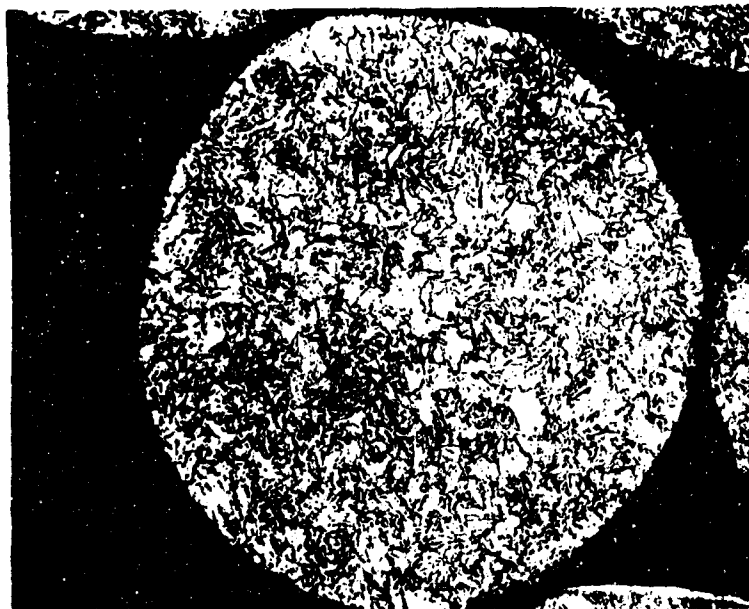
made on the as-received conductor, after exposure to potassium vapor at 850 C. and vacuum-annealed at 850 C. The Knoop hardness values at a 300 gm loading were: 431 for the as-received rhodium conductor, 172 after 850 C potassium vapor exposure and 248 after a four-hour vacuum anneal at 850 C. Micrographs of rhodium wire are shown in Figures 36, 37 and 38.

The exposure of rhodium wire to potassium vapor for 172 hours at 850 C and vacuum annealing at 850 C for 4 hours increased the ductility and grain size and decreased the hardness as compared to as-received rhodium wire.

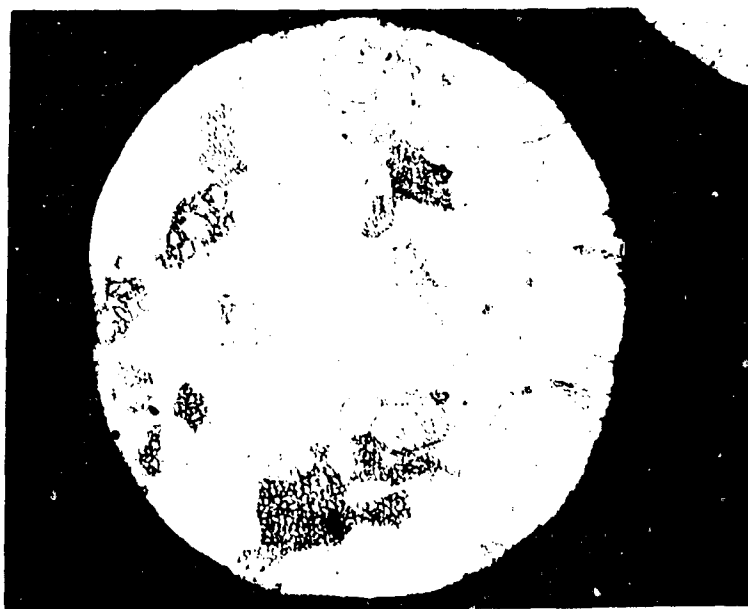
#### E. Transformer Fabrication and Electrical Testing

##### 1. Preliminary Test Transformer - Electrical Testing

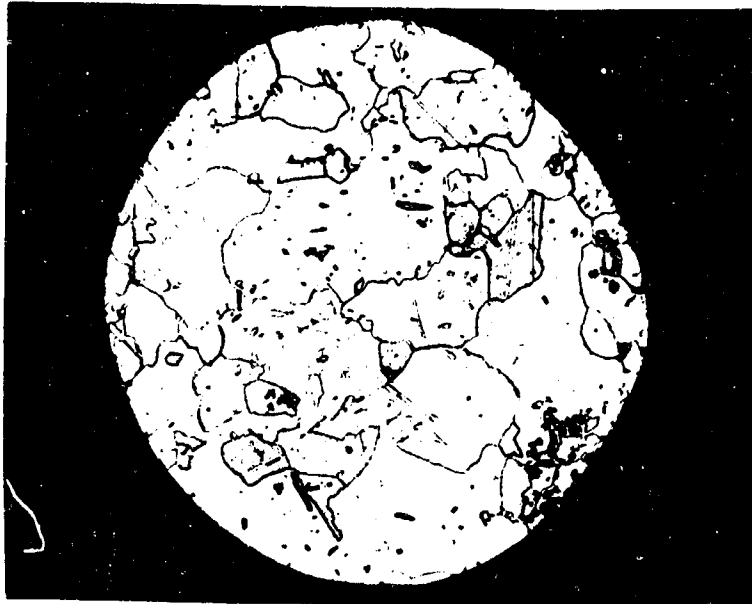
The preliminary test transformer described in the Second Quarterly Report, consisting of 50 turns of No. 22 AWG wire primary and 5 turns of No. 6 AWG wire secondary, was canned in a stainless steel test capsule with an electrical terminal seal. The test capsule containing the transformer and 0.8 ohm resistive load, shown in Figure 39, was placed in an inert atmosphere (argon) furnace. A separate heater was installed in the furnace to heat the electrical terminal seal, thus preventing condensation of potassium vapor on the ceramic portion of the terminal seal.



**FIGURE 36.** Rhodium Wire, as-received; 0.0254 inch O. D.  
Electrolytic Etch, Conc. HCL (100X)

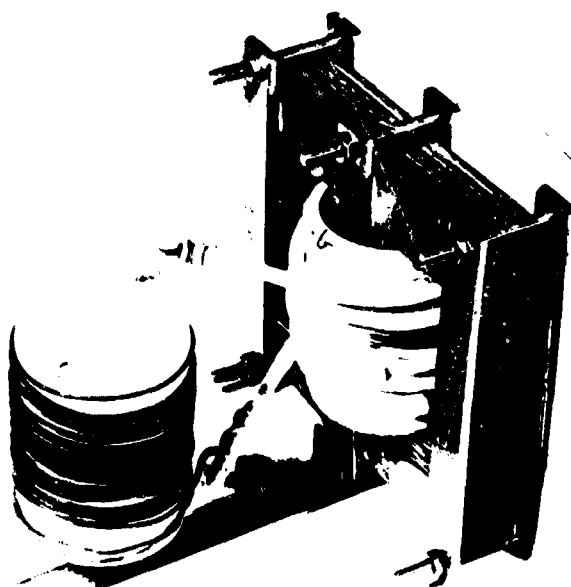


**FIGURE 37.** Rhodium Wire, After Exposure to Potassium  
Vapor @ 850 C for 172 Hours; 0.0254 inch O. D.  
Electrolytic Etch, Conc. HCL (100X)



**FIGURE 38. Rhodium Wire, Vacuum Annealed @ 850 C for 4 Hours; 0.0254 inch O. D. Electrolytic Etch, Conc. HCL (100X)**

**WAED64. 75E-67**



**FIGURE 39. Preliminary Test Transformer - Plasma Arc  
Sprayed Alite A-610 (99%  $\text{Al}_2\text{O}_3$  - 1%  $\text{MgO}$ )  
Insulation and Insulators - With 0.8 Ohm  
Resistive Load**

**WAED64. 75E-68**

Condensation of potassium vapor on this seal may occur if the capsule temperature is higher than the seal temperature. This preliminary test transformer was designed to give data on electrical tests in potassium vapor at elevated temperature and indicate potential problem areas before proceeding with a more sophisticated test transformer.

a. Electrical Testing in Argon at 23, 300 and 600 C Ambient

The argon furnace was evacuated and flushed with argon containing < 3 PPM oxygen. An argon atmosphere at 3 psig was used to protect the columbium - 1% zirconium electrical terminal seal.

The transformer was energized by applying various a-c voltages to the 50 turn primary winding at a 400 cps frequency. The corresponding current values from these voltages were recorded and plotted to obtain load curves at the varying temperatures. The nichrome wire resistive load varied from 0.8 ohm at 23 C to 0.86 at 600 C. Figures 40, 41 and 42, respectively, show how the electrical input requirements of the primary varied with temperature. These experiments were run so effects due to temperature could be subtracted from effects due to temperature plus potassium vapor.

b. Electrical Testing in Potassium Vapor at 23, 290, 330, and 450 C

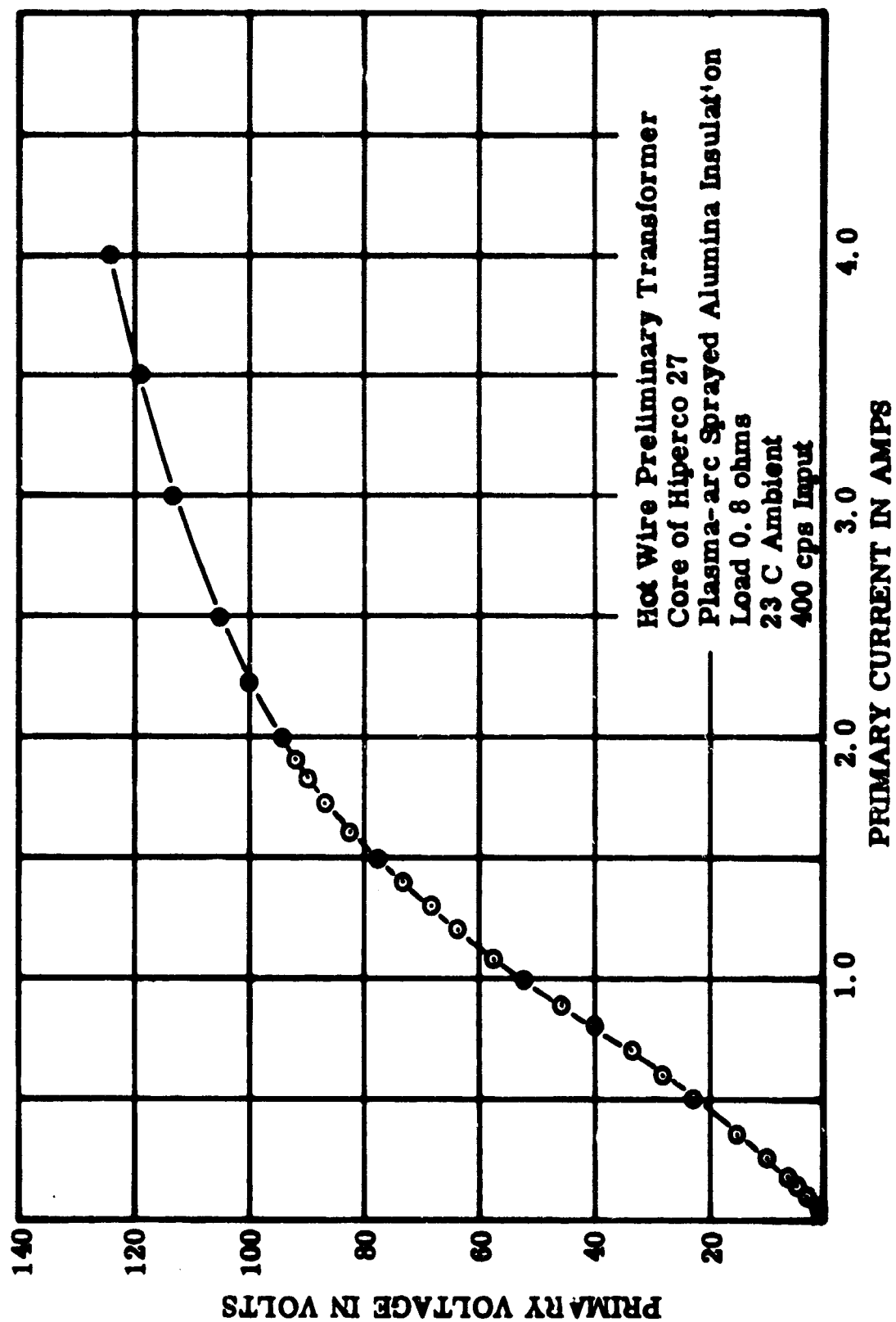


FIGURE 40. Primary Load Curve - Preliminary Test Transformer in Air Room Ambient Temperature - 400 cps Frequency

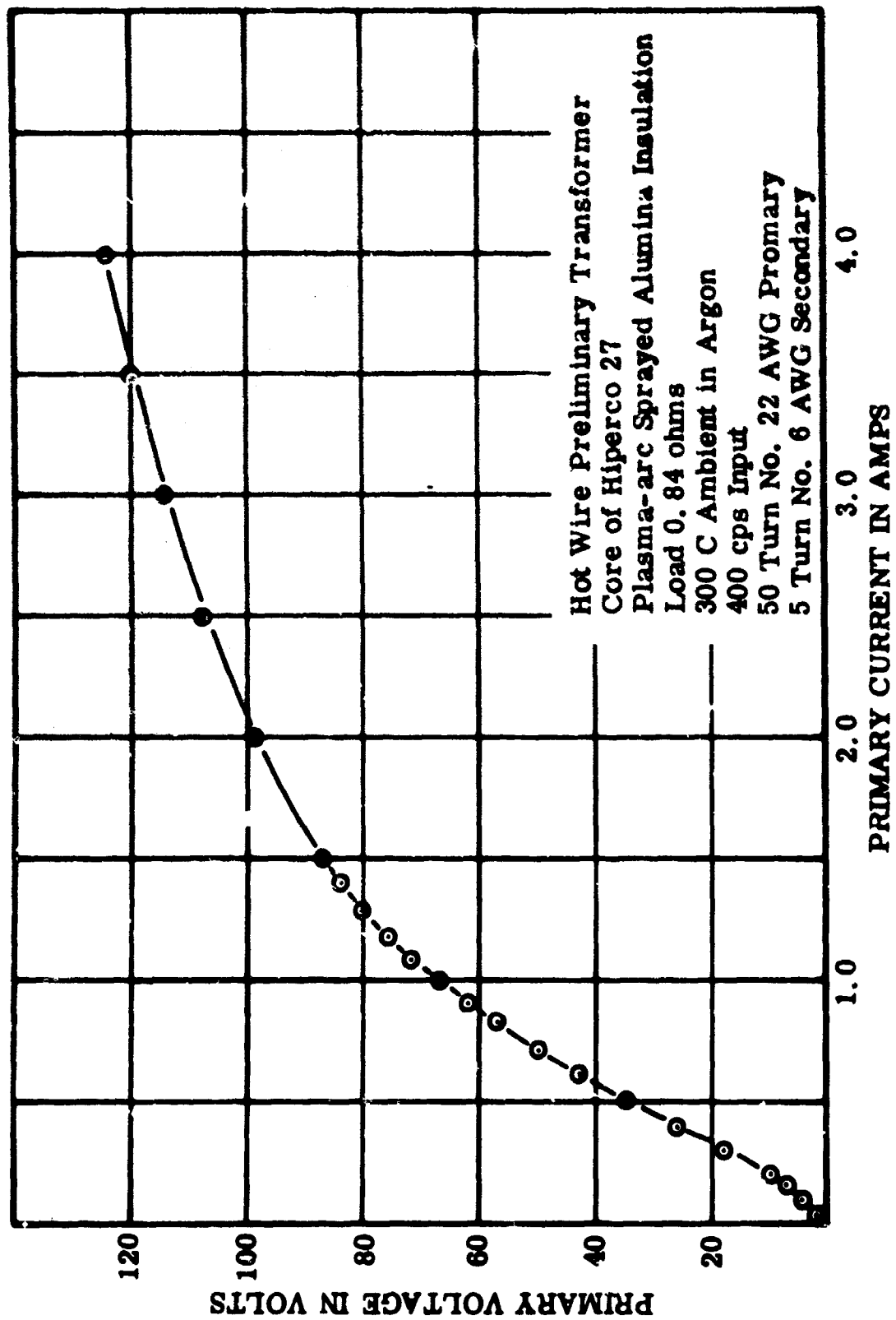


FIGURE 41. Primary Load Curve - Preliminary Test Transformer in a Room at 300 C - 400 cps Frequency

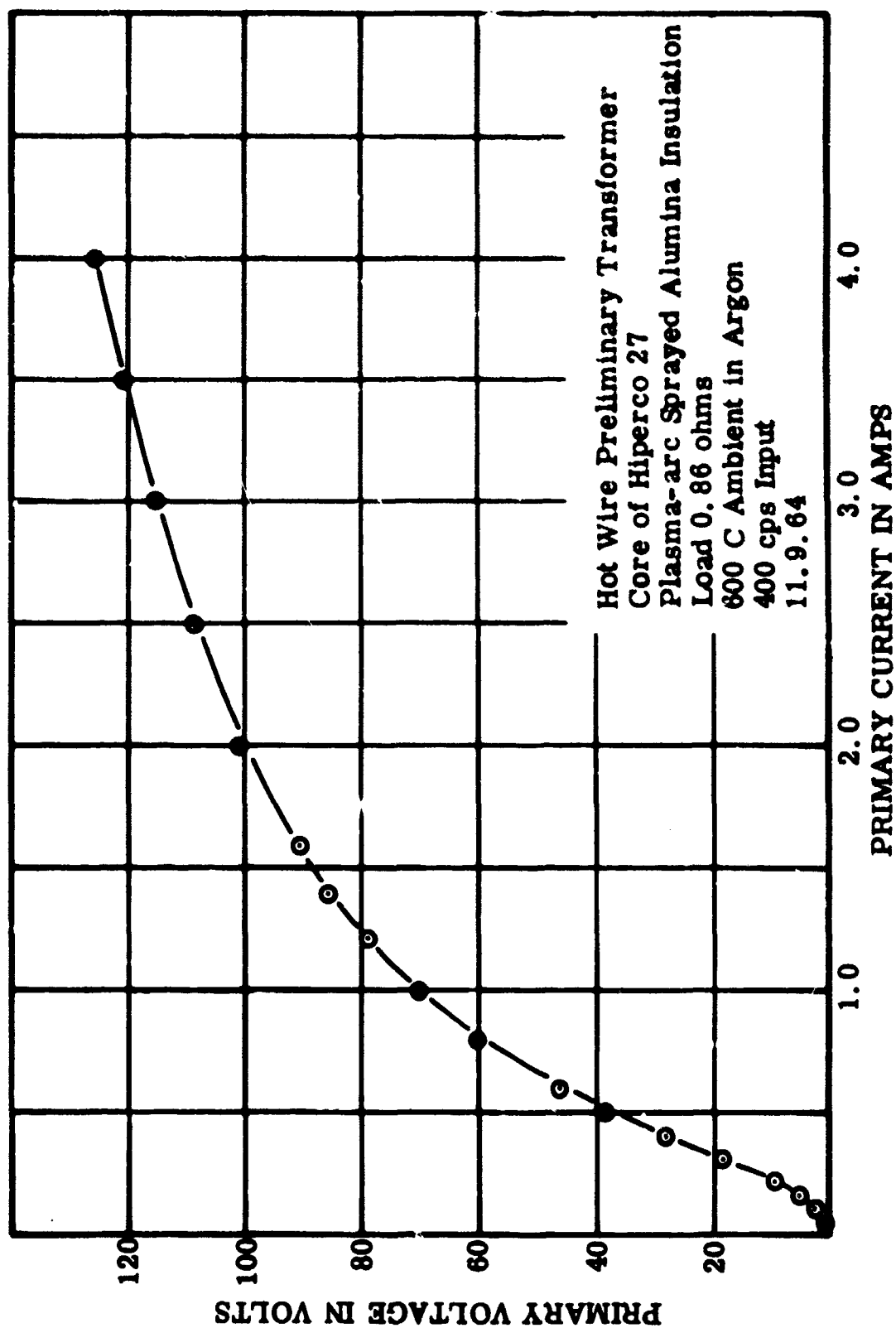


FIGURE 42. Primary Load Curve - Preliminary Test Transformer  
 in Argon at 600 C - 400 cps Frequency

A 20 gram quantity of purified potassium containing  $< 50$  PPM oxygen was introduced into a test capsule via a stainless steel fill tube. The capsule was then connected to a high vacuum line ( $10^{-5}$  torr) and evacuated. All the loading and other manipulations were done in an argon-filled glove box with a measured oxygen concentration of  $< 3$  PPM.

The vacuum line was also inside this glove box.

After the capsule was cut from the vacuum line and welded shut, it was placed in a vacuum furnace. The transformer was energized at an ambient temperature of 23 C. The temperature of the transformer went to 93 C and data points taken were again plotted. At a reading of 120-130 volts a-c and 3.5-4.0 amperes, an arcing noise was heard. The applied test voltage was immediately switched off and the test was stopped. The test was resumed again after a waiting period of approximately two minutes. The second set of data points varied considerably from the first set and the temperature was lower (maximum of 49 C). Figure 43 illustrates the difference in input voltage and amperage. Resistance readings, taken across the primary coil after testing, indicated no change in the d-c resistance ( $\sim 10$  ohms).

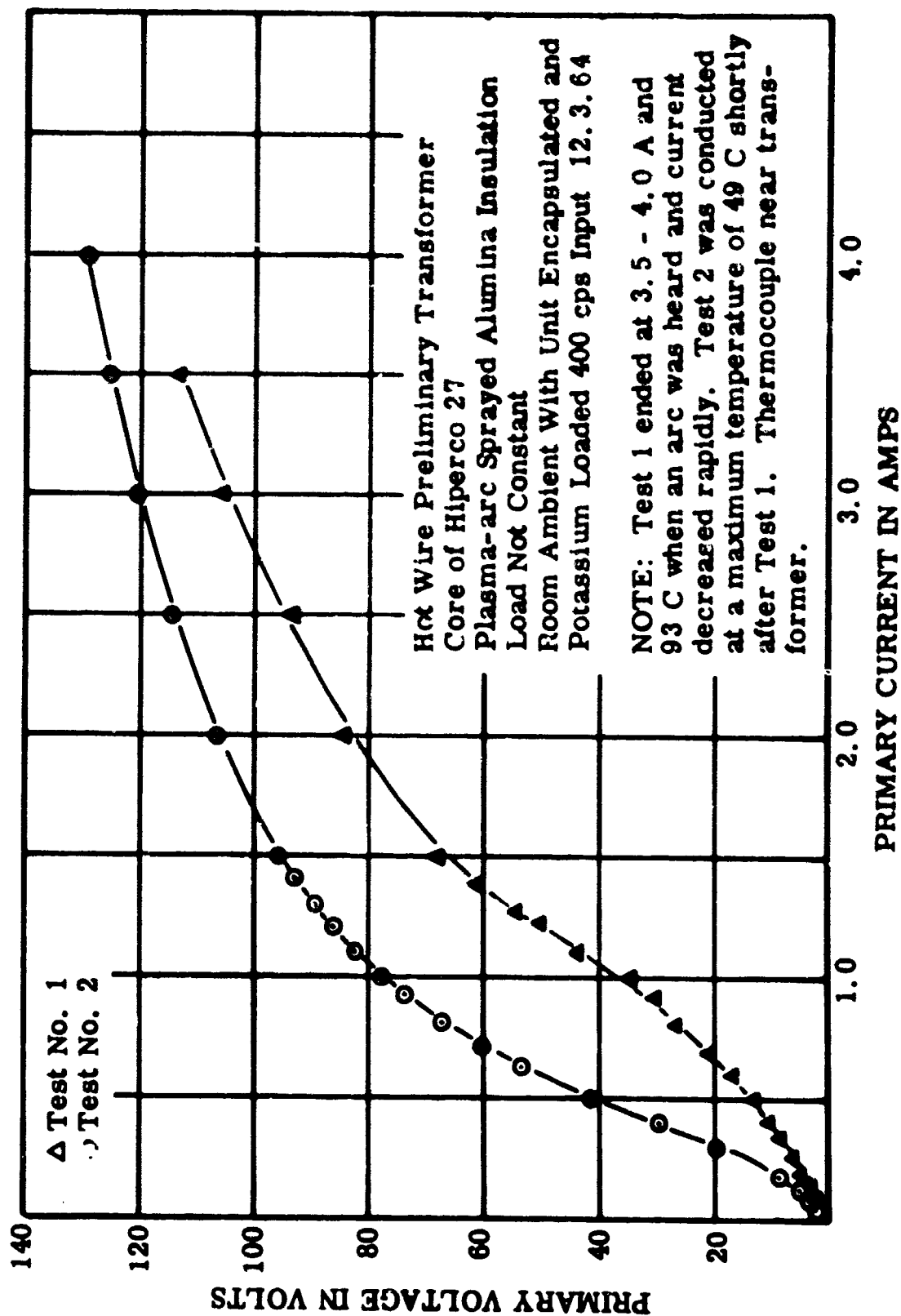


FIGURE 43. Primary Load Curves - Preliminary Test Transformer in Saturated Potassium Vapor at 93 and 49 C

The transformer and capsule were allowed to cool after testing at 93 and 49 C before going to the higher temperatures. Potassium condensed on the transformer and the ceramic-to-metal seal causing a direct short. The short opened at 116 C and tests were run at 290, 333, and 450 C. Figure 44 shows a comparison of load data taken at these temperatures.

A separate heater surrounding the electrical terminal seal was used on the first tests to prevent condensation. For the last three test temperatures, however, this heater was deliberately not used. This was done to determine the maximum temperature differential that could exist between an electrical terminal seal and a device electrically energized and operating in a saturated potassium vapor.

When the terminal seal temperature was ~377 C and the transformer temperature reached 527 C, a short circuit developed blowing the fuse in the ammeter. The fuse was replaced and the terminal seal temperature increased slowly. An indicator current of one ampere was used to tell when the shorted terminal seal would again have a high electrical resistance. At 15 C over the transformer temperature of 527 C, the short circuit opened; however, the transformer could not be energized as had been done prior

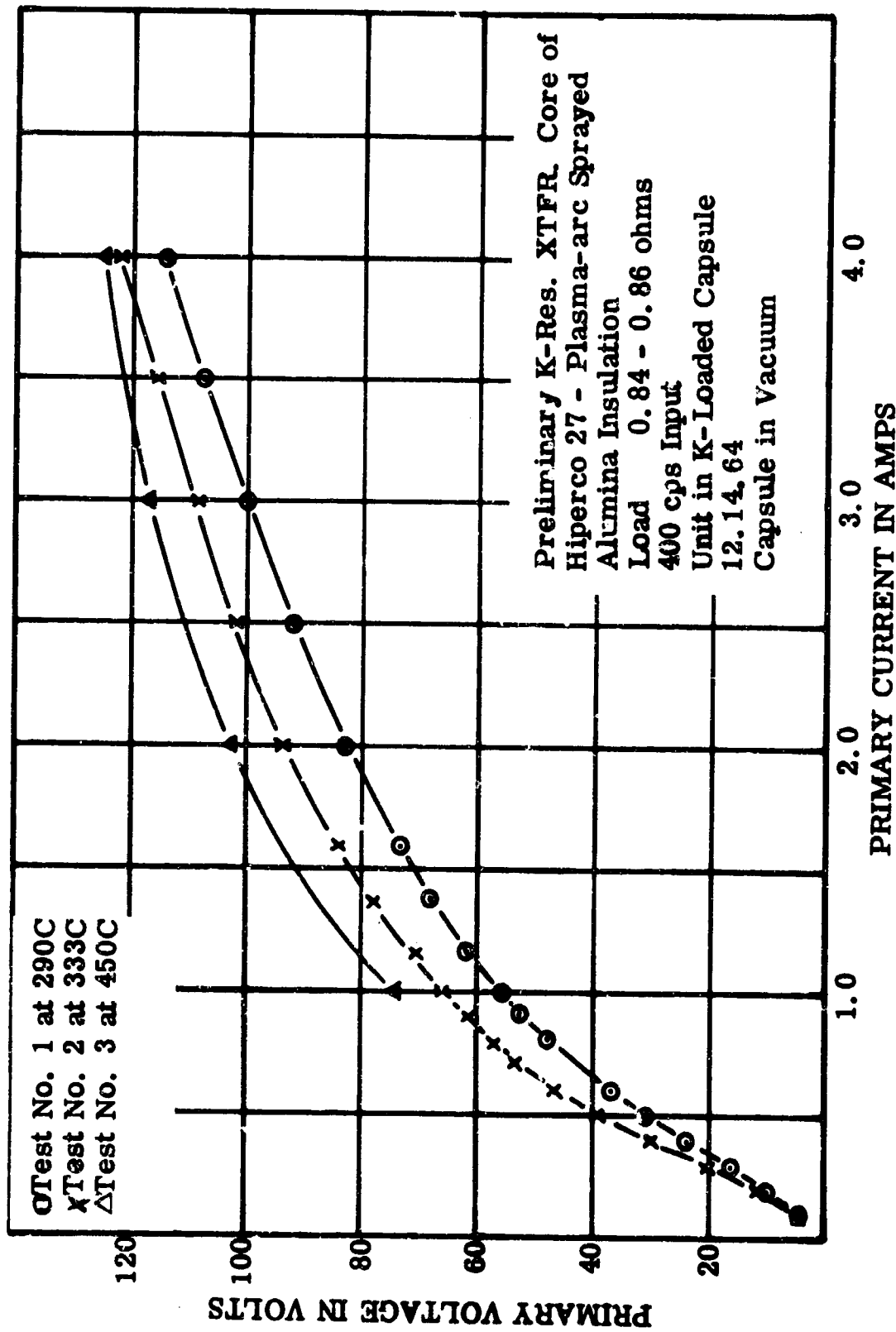


FIGURE 44. Primary Load Curves - Preliminary Test Transformer  
 in Saturated Potassium Vapor at 290, 333, and 450 C

to this short circuited condition. Resistance reading of 35,000 ohms instead of 10 ohms across the primary indicated an open condition existed.

The furnace was cautiously opened but no potassium leaks had occurred and the terminal seal was intact. Visual examination of the position of the primary lead wire to the terminal seal by means of an intense light beam transmitted through the translucent Lucalox portion of the seal indicated the primary transformer lead may have come loose from the crimped portion of the terminal seal.

The operation of a transformer in saturated potassium vapor appears possible at the condition of 120 volts a-c, 4 amperes, 400 cps, and 450 C based upon the cursory data available to date.

## 2. Ceramic Coil Form Transformer

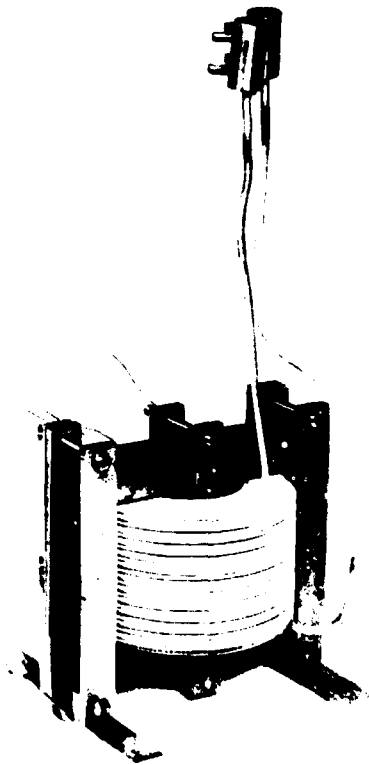
### a. Fabrication

The construction of the ceramic coil form transformer is shown in Figures 45 and 46. The primary coil forms have 515 turns of No. 23 AWG nickel-clad silver. They were designed to be wound with a continuous winding of conductor and to slip one inside the other when wound. The secondary winding has 11 turns of No. 8 AWG nickel-clad silver that has been plasma-arc sprayed with approximately three mils



**FIGURE 45. Ceramic Coil Forms Wound and Assembled -  
Primary Coil Forms and Secondary Coil Form**

**WAED64. 75F-78**



**FIGURE 46. Ceramic Coil Form Transformer**

**WAED64. 75E-79**

of nickel aluminide followed by two mils of high-purity alumina (Linde A 99.99%  $\text{Al}_2\text{O}_3$ ). The core was fabricated from 0.008 inch Hiperco 27 with a plasma-arc sprayed alumina insulation.

b. Electrical Testing at Room Temperature

The transformer was assembled and preliminary tests were made to determine its characteristics. Short circuit and open circuit tests were made to show variations as input voltage and frequency were varied. Table 6 shows this data.

With the conditions specified in the contract, the maximum flux is obtained at the maximum input voltage of 1000 volts and the minimum frequency of 400 cps.

The short circuit tests, with 50 amperes short circuit current, show that the voltage required to overcome the leakage inductance is approximately 75 volts at 400 cps. It increases as expected almost proportionally with frequency to about 180 and 540 volts at 1000 and 3200 cps respectively. Short circuit tests are generally made at conditions where there are little or no losses other than the conductor. The characteristics of the transformer are such that the flux in the core remains almost constant as the frequency increases.

**TABLE VI. Ceramic Coil Form Transformer - Electrical Test Results  
at Room Temperature**

**Short Circuit Test**

<b>Frequency (cps)</b>	<b>*Input Voltage</b>	<b>Input Amps</b>	<b>Input Watts</b>	<b>Based on 1000 V Input</b>	
				<b>Impedance (Percent)</b>	<b>Leakage Induc- tance (Percent)</b>
400	86	1.06	44.8	8.6	7.5
1000	190	1.06	64	19.0	18
3200	560	1.06	155	56	54

**Open Circuit Test**

<b>Frequency (cps)</b>	<b>Excitation Voltage</b>	<b>Excitation (Volt-Amperes)</b>	<b>Core Loss (Watts)</b>
400	100	2.43	1.6
	500	44.2	27.0
	1000	418.0	102.0
1000	100	1.48	0.8
	500	23.3	16.4
	1000	80.3	61.5
3200	100	-	-
	500	13.45	9.5
	1000	44.3	37.0

\*The input voltage shown at short circuit conductors is that required to overcome the transformer impedance, causing 50 amperes to flow in the short circuited winding.

The flux level is low enough that, at 400 cps, little or no core loss is present.

At 1000 and 3200 cps, there is more core loss present which would account for the deviation of the impedance volts from the proportional.

The short circuit tests show that a load current of 50 amperes cannot be produced with 1000 cps - 100 volts, or 3200 cps, - 100 and 500 volts output. Tests also show there is considerable increase in conductor losses as the frequency increases. The increase in losses may be caused by higher eddy current losses, by skin effect of the current, by the higher resistance of the conductor surface, or by any or all combinations.

The open circuit tests show the core loss is in the expected range and that the interlaminar insulation is satisfactory.

The core loss is high at the maximum flux condition, but other test data indicate the loss will be reduced to about one-half at 600 C.

A temperature rise test was made at room ambient, 1000 volt, and 400 cps input condition. The temperature rise was determined by the change in resistance of the input winding. The temperature rise was 125 C. The input and

output parameters were also measured at the start and finish of the test. At approximately a 50 C temperature condition, the transformer output was 950 voltamperes at 50 amperes and the input was 1280 voltamperes. Just prior to removal of power to the unit, the output was 940 voltamperes at 50 amperes load.

Performance tests at 500 volt 400 cps input condition resulted in 442.5 voltamperes output at 50 amperes with 580 voltamperes input.

## IV. FUTURE WORK

### A. Ceramic-to-Metal Seals

Attempts will be made to degas Cb-Al<sub>2</sub>O<sub>3</sub> concentric ring seals in a cold-wall vacuum furnace at 1300-1700 C. The oxygen content of the columbium is of importance because of its possible detrimental effect on resistance to corrosion in saturated potassium at 600-850 C.

Hardness tests will be made on the columbium portion before and after vacuum degassing. In addition, microstructural studies and leak tests will be performed.

Additional Cb-Al<sub>2</sub>O<sub>3</sub> concentric ring seals will be hot pressed in order to provide at least four seals which can be used for hermetic, potassium resistant electrical feed-throughs for a test on an encapsulated high temperature transformer. Cb-1Zr wire will be used for the electrical conductor. The seals will be brazed inside one end of a 3 inch length of type 316 stainless steel pipe and/or Kovar tubing.

Nicrobraz 130 will be the brazing alloy. The other end of the pipe or tubing will be TIG welded to the stainless steel test capsule. Preliminary grinding, machining, drilling, and tapping evaluations will be undertaken on the refractory metal portions of Ta-Al<sub>2</sub>O<sub>3</sub> and Cb-Al<sub>2</sub>O<sub>3</sub> seals to determine beforehand suitable procedures for the final seals.

Consideration will be given to working out a technique for mixing zirconium metal powder with the columbium before compacting in order to introduce a getter which would be expected to lower the oxygen content of the columbium metal in the seal.

**B. Magnetic Materials**

Room temperature tests are to be completed on Rowland rings of 5% Al-Fe and 3% Al, 1% Y-Fe alloys. Metallographic examination of all samples will be completed to check for corrosion, and also brittleness checks will be made.

**C. Conductor Insulating and Transformer Fabrication**

The No. 22 columbium-clad dispersion-strengthened copper will be used to fabricate an insulated transformer primary coil. This coil will be either plasma-arc sprayed with alumina while winding or dip coated, wound green, and fired. The fluoride eutectic potting compound will be used to pot this transformer.

**D. Transformer Testing**

The transformer described above will be put in an electrical test capsule with purified potassium and tested after first obtaining operating characteristics at 600 C in argon. The ceramic coil form transformer will be encapsulated and tested at 600 C under the following electrical conditions, first in argon or vacuum, then in potassium.

## Electrical Test Conditions

### Primary

<u>A-C Voltage</u>	<u>Frequency CPS</u>	<u>Temperature</u>
100	400	600 C
500	400	600 C
1000	400	600 C
100	1000	600 C
500	1000	600 C
1000	1000	600 C
100	3200	600 C
500	3200	600 C
1000	3200	600 C

If these conditions are satisfactorily met in potassium vapor, the transformer will be life tested for 500 hours at 500 Vac and 1000 cps.